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BULLETIN No. 43.

U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

PROCEEDINGS

OF THE

ELEVENTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

WASHINGTON, D. C.,

AUGUST 23, 24, AND 25, 1894.

EDITED BY

HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.

PUBLISHED BY AUTHORITY OF THE SECRETARY OF AGRICULTURE.

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LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Division of Chemistry,
Washington, D. C., September 14, 1894.

Sir: I transmit herewith for your inspection and approval the manuscript containing the proceedings of the eleventh annual meeting of the Association of Official Agricultural Chemists, and ask that it be published as Bulletin 43 of the Division of Chemistry of this Department.

H. W. Wiley,
Chief of the Division of Chemistry and Secretary of the Association of Official Agricultural Chemists.

Hon. Chas. W. Dabney, Jr.,
Acting Secretary.

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PROCEEDINGS OF THE ELEVENTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, HELD AT WASHINGTON, D. C., AUGUST 23, 24, AND 25, 1894.

FIRST DAY.

THURSDAY—MORNING SESSION.

The members assembled in the lecture room of the National Museum at 10 o'clock a. m. August 23. In the absence of the president, E. B. Voorhees, the vice-president, H. A. Huston, occupied the chair. The secretary read a telegram from Mr. Voorhees, stating that he was unavoidably detained, but hoped to be present on the following day. The presiding officer, by vote of the association, appointed a committee on the order of business, consisting of B. B. Ross, W. A. Rawls, and H. B. McDonnell. The presiding officer also appointed a committee to call on the Assistant Secretary of Agriculture and ask him to attend the meeting. The committee consisted of J. T. Anderson and George F. Payne.

The president's address was read by the acting president as follows:

ADDRESS OF THE PRESIDENT.

This marks the beginning of our eleventh year as a working association, and we have good reason to congratulate ourselves on our progress.

It is not my purpose to review in detail our past history, which speaks for itself in our published records, nor to weary you with platitudes in regard to the possibilities of our future. It seems to me, however, eminently proper at this time—the beginning of the second decade of our existence as an association—that attention should be called to certain features of our development, and to the lines of influence and usefulness that may be exerted by such an organization as ours.

In this age of material progress, conventions, associations, combinations, annual meetings, etc., are the order of the day; the organization or combination is what counts and not the individual. This affects, too, all classes and lines of work—industrial, social, political, educational, and scientific. These various forces are all striving for recognition, and there is danger that the primary object of any of them may be forgotten in the haste for position and influence in the little world that knows them.

We must not forget our beginnings, and also that progress is not measured by forward movement, except that in the movement there is an actual gain in result in line with the primary object of the organization.

In May, 1884, a few of our leading agricultural chemists, chiefly those charged with the official control of the sale of fertilizers in their respective States, met in Atlanta to devise ways and means for the improvement of analytical methods. The outcome of that meeting, though not the first of its kind, is our present association, the objects of which at first, as defined by the constitution then adopted, were "to secure as far as possible uniformity in legislation with regard to the regulation of the sale of commercial fertilizers in the different States, and uniformity and accuracy in the methods and results of fertilizer analyses."

In 1885, in accordance with the first clause of this section of the constitution, a committee was appointed to consider the question of uniformity of legislation and regulations as to the inspection of fertilizers, and to prepare a draft of a uniform law to be submitted to a convention, to be held at Washington, to consider the matter. A careful study of the proceedings of the association fails to reveal the report of this committee.

In the proceedings for 1886, however, in reply to a communication from the secretary of the National Fertilizer Association urging the coöperation of this association in the abolishment of commercial valuations, strong ground is taken by prominent members against our taking part in any national legislation. This was followed in the same year by a change in our constitution, which eliminated this clause and thus relieved the association from taking a definite position on an important matter, and one then believed to be extremely difficult of adjustment, though it was recognized to intimately concern members of the association and to directly affect an enormous industry, the right development of which is, either with or without our assistance, vital to our agricultural interests.

It was perhaps the part of wisdom for the association at that time to amend its constitution in this particular, and to confine its work to matters of a purely scientific character, and upon which we could in time all agree, at least in essentials.

Careful study of the status of our association at the present time, after great things have been accomplished in the line of accuracy and uniformity of agricultural chemical methods, convinces me that it is entirely within our province to at least discuss these points, if not to take positive ground in reference to those questions first regarded as of primary importance in respect to the fertilizer trade, and now, by virtue of their development, extended to various other lines of industry which concern the official chemist.

This is still provided for by part 2, section 1, of the constitution, which reads as follows: "To afford opportunity for the discussion of matters of interest to agricultural chemists." It is of interest to agricultural chemists to see to it that the laws enacted in the various States for the control of the sale of those products, which we as official chemists must aid in executing, should be just to both producer and consumer, that protection should protect both the honest and good manufacturer and the ignorant and careless consumer, and that time and money should not be spent annually in unnecessary and meaningless analyses.

We are an association of agricultural chemists, not mere analysts, and are, or should be, interested in the far-reaching influence of a proper chemical analysis.

The fertilizer industry alone has doubled in volume of business and capital invested in the last decade, with a proportionate increase in use, due in no small degree to the information given by agricultural chemists, and the influence of this Association of Official Chemists should be a power on the side of honesty and righteousness in this development, not only in the uniformity and accuracy of the chemical results secured, but in showing the usefulness of the analyses made, and in making the publication of the results. at least, indicate very closely the value of the materials examined.

Under the conditions that exist at present the various chemists must stand credited with a wide diversity of views in reference to the meaning and value of an analysis, as indicated by their published results, whether they so will or not, simply because they are engaged in executing the law in force in their respective States, and thus, in many cases. helping to perpetrate a series of absurdities upon a confiding public. It seems to me that it is proper that we should decide whether we can afford to stand so credited.

I speak freely on this matter, with the full knowledge that my own State is the chief of sinners in this respect. It is not my purpose in these remarks to detract from the value of the work done by the association in specific lines, particularly in the improvement of analytical methods, but rather to emphasize its importance by contrasting our progress in this direction with the lack of development in other directions intimately connected with it.

The proceedings of the first meeting are also interesting from another standpoint, viz, the prominence then given to the discussion of the methods of analysis of phosphates, particularly that form of phosphoric acid called "reverted," evidently regarded at that time as a mythical something without form or substance.

Five-sixths of the report of the meeting are given up to a discussion of this one point, while the methods of analyses for nitrogen and potash receive but a passing notice.

A careful study of our records for the past ten years shows that the importance of this question continues to be recognized, and while we have cause for great encouragement there is still much to be desired, both in the way of uniformity and accuracy of results.

There is perhaps no one thing that gives so much anxiety to the average agricultural chemist as this determination of the "available." A series of concordant results obtained on a difficult product is to him like the elixir of life, though the exhilaration is short-lived, for he lies down at night with the fear that in the morning he may find that a product has been discovered which, while showing remarkable results in the field, does not respond equally nor uniformly to his test.

If we attempt to show the percentage of "available" by our chemical methods, these methods must keep pace with progress in other directions, for although it has been wisely remarked that "there is nothing new under the sun," that was strictly true for the term "available" under the old dispensation, or when boneblack was the chief source of superphosphate.

We hear much in these days, too, of "available" nitrogen. In the good old times the determination of total nitrogen was sufficient, and even now many of us are satisfied with the separation of the nitrogen into the three forms—nitrates, ammonia salts, and organic—knowing full well that the latter term, particularly in case of mixed goods may, and frequently does, cover a multitude of sins.

Our future work must include a study of methods that shall enable us to at least give some idea of the source and availability of the organic nitrogen in mixed fertilizers.

In reference to fodder analyses I can only emphasize the suggestions made by Dr. Babcock last year, viz, "to substitute for the present illogical and unmeaning system one which, so far as it went, would deal with the proximate constituents which the fodders contain."

In response to these suggestions, and the strong reasons for their support, considerable effort has been expended this year in the right direction, and the association should in every legitimate way show its appreciation by a continued and growing interest in support of this line of work.

The lines of development and increasing usefulness of our association is, perhaps, made sufficiently clear by the hints already given, though not by any means confined to the points mentioned. Our field includes all branches of agricultural chemistry, and as the various methods are perfected the necessity for investigation arises, work that requires not only the highest skill in manipulation, but a thorough knowledge of the whole field of agricultural science, and an almost infinite patience and persistence in the work. Just here a serious difficulty arises; many of our members are so situated as to be unable to give the time or provide for the expenses of the investigation.

The governing boards of our various institutions in too many cases do not appreciate the value of investigations in this direction; some, indeed, measure the value of the work of their chemists by the number of analyses performed, or the number of pages of the bulletins issued, and hence they are forced to do the association work either in a hurry or in their own time, and at their own expense. Under the circumstances, therefore, while many make self-denying efforts and do most excellent work, the reporters find that if real investigation is required it is too often pursued as far as the sailing is smooth, and the remainder is left undone, with the lame excuse "that time at their disposal will not permit."

In order to avoid this difficulty, either the workers must be limited still further in the number of actual investigations required of them, or ways and means must be provided whereby those willing to do the work may be enabled to pursue it and those unwilling may be made willing. It has been suggested that a slight appropriation is most admirably suited to effect a change of opinion in this respect. I am satisfied that the question of how the work shall be done in the future merits serious consideration at this time.

Another feature of our work seems, too, to be a matter of congratulation; I refer to the coöperation of foreign chemists.

In accordance with the suggestions of the resolutions passed at our last annual meeting, the following action was taken by your executive committee:

1. That the secretary of the association, Dr. H. W. Wiley, chemist of the U. S. Department of Agriculture, communicate the action of the association to the leading chemists and chemical associations of foreign countries.
2. That the secretary incorporate in said communications an outline of the method of the work adopted by this association, and to invite coöperation, as far as possible, in the work of the present year.
3. That in view of this action, if foreign chemists express a willingness to take part in the work, they shall, upon application to the secretary, Dr. H. W. Wiley, be furnished with samples and the accompanying directions necessary to properly carry it out.

Dr. Wiley has faithfully performed his duty in this matter, over two hundred and fifty leading foreign chemists having been invited to coöperate with us.

Thus the field of usefulness of our association, outgrowing in the first decade of its existence the confines of our own broad country, imposes upon each one of us a work of grave responsibility, both in maintaining our past reputation and increasing the efficiency of our work, a realizing sense of which should be shown in our deliberations here, as well as in the investigations conducted in our own laboratories throughout the coming year. [Applause.]

At the conclusion of the reading of the address, the committee on order of business reported as follows:

PROGRAMME.

1. Appointment of a committee of five on recommendations of reporters.
2. Report on dairy products.
3. Report on potash.
4. Report on soils and ash.
5. Report on phosphoric acid.
6. Report on sugar.
7. Report on nitrogen.
8. Report on cattle foods.
9. Report on fermented liquors.
10. Report of abstract committee.

Election of officers to be the special order for Friday, 24th instant, at 3 p. m. Hours for meeting, 9 a. m. to 12:30 p. m.; 1:30 p. m. to adjournment.

B. B. Ross.
H. B. McDonald.
W. A. Rawls.

The report of the committee was adopted and the committee discharged.

The following officers and members of the association were in attendance:

MEMBERS AND VISITORS PRESENT.

President, E. B. Voorhees, Director, Agricultural Experiment Station of New Jersey, New Brunswick, N. J.

Vice-President, H. A. Huston, State Chemist of Indiana, Lafayette, Ind.

Secretary, H. W. Wiley, Chief of Chemical Division, U. S. Department of Agriculture, Washington, D. C.

Member of executive committee, B. B. Ross, State Chemist of Alabama, Auburn, Ala.

Allen, E. W., U. S. Department of Agriculture, Washington, D. C.
Anderson, James, Assistant Chemist, Agricultural Experiment Station, Auburn, Ala.
Bartlett, J. M., Chemist, Maine Experiment Station, Orono, Me.
Beal, W. H., Office of Experiment Stations, U. S. Department of Agriculture, Washington, D. C.
Bigelow, W. D., Assistant, Chemical Division, U. S. Department of Agriculture, Washington, D. C.
Bromwell, William, Johns Hopkins University, College Park, Md.
Carman, J. S., Assistant, Chemical Division, U. S. Department of Agriculture, Washington, D. C.
Carpenter, F. B., Assistant Chemist, North Carolina Experiment Station, Raleigh, N. C.
Carr, Oma, Assistant, Chemical Division, U. S. Department of Agriculture, Washington, D. C.
Chute, H. D., Chemist, Cherrydale, Va.
Crampton, C. A., Chemist, Internal Revenue, Washington, D. C.
Cutter, W. P. (late Chemist Utah Station), Librarian, U. S. Department of Agriculture, Washington, D. C.

Davidson, R. J., Chemist, Virginia Experiment Station, Blacksburg, Va.

Farrington, E. H., Dairy Husbandry, Madison, Wis.

Finney, F. I., Chemist, Savannah, Ga.

Frear, William, Vice-Director, Pennsylvania Experiment Station, Board of Agriculture, State College, Pennsylvania.

Fueiling, J. L., Chief Chemist, Brooks & Co., St. Jago, Cuba.

Gascoyne, W. J., private laboratory, Baltimore, Md.

Herff, B. von, The Chemist Representative of the German Kali Works, 99 Nassau street, New York.

Kedzie, F. S., Adjunct Professor of Chemistry, Michigan Agricultural College, Agricultural College, Mich.

Kepich, A., Chemist of Elk Tanning Company, Ridgway, Pa.

Kerr, G. A., Chemist of Mooney & Son, tanners, Columbus, Ind.

Kilgore, B. W., Assistant Chemist, North Carolina Experiment Station, Raleigh, N. C.

Krug, Wm. H., Assistant, Chemical Division, U. S. Department of Agriculture, Washington, D. C.

Lindsey, J. B., Chemist of Massachusetts State Experiment Station, Amherst, Mass.

McDonnell, M. S., Pennsylvania Agricultural Experiment Station, State College, Pa.

McDonnell, H. B., State Chemist of Maryland, College Park, Md.

McElroy, K. P., Second Assistant Chemist, Chemical Division, U. S. Department of Agriculture, Washington, D. C.

Morse, Fred., Chemist, New Hampshire Experiment Station, Durham, N. H.

Nolan, W. P., American Fertilizer Company, Philadelphia Pa.

Patterson, H. J., Chemist, Maryland Agricultural Experiment Station, College Park, Md.

Payne, George F., State Chemist of Georgia, Atlanta, Ga.

Peacock, S., American Fertilizer Company, Philadelphia, Pa.

Perkins, W. R., Chemist, Missouri Experiment Station, Agricultural College, Mo.

Peter, A. M., Chemist, Kentucky Agricultural Experiment Station, Lexington, Ky.

Rawls, W. A., State Chemist, of Florida, Tallahassee, Fla.

Roode, R. de, Chemist, West Virginia Agricultural Experiment Station, Morgantown, W. Va.

Runyan, E. G., Assistant, Chemical Division, U. S. Department of Agriculture, Washington, D. C.

Sanborn, T. F., Assistant, Chemical Division, U. S. Department of Agriculture, Washington, D. C.

Schweinitz, E. A. de, Chemist, Biochemic Laboratory, U. S. Department of Agriculture, Washington, D. C.

Sherman, H. C., Agricultural College, Maryland, College Park, Md.

Shuttleworth, A., Professor of Chemistry, Ontario Agricultural College, Gueiph, Canada.

Spencer, G. L., First Assistant, Chemical Division, U. S. Department of Agriculture, Washington, D. C.

Steiger, G., U. S. Geological Survey, Washington, D. C.

Street, J. T., Assistant Chemist, New Jersey Agricultural Experiment Station, New Brunswick, N. J.

Tassin, Wirt, U. S. National Museum, Washington, D. C.

Terne, B., Chemical Manager Delaware River Chemical Works, Philadelphia, Pa.

Test, F. C., Office of Experiment Stations, U. S. Department of Agriculture, Washington, D. C.

Van Slyke, L. L., Chemist, New York Experiment Station, Geneva, N. Y.

Veithch, F. P., Assistant Chemist, Maryland Agricultural College, College Park, Md.

Voorhees, L. A., Chemist, New Jersey Agricultural Experiment Station, New Brunswick, N. J.

Woll, F. W., Assistant Chemist, Wisconsin Experiment Station, Madison, Wis.
Watson, T., Assistant Chemist, Virginia Experiment Station, Blacksburg, Va.
Westenfelder, B., Chemist, American Oak Leather Company, Cincinnati, Ohio.
Wheeler, H. J., Chemist, Rhode Island Agricultural Experiment Station, Kingston, R. I.
Whitney, M., Chief of Division of Agricultural Soils, U. S. Department of Agriculture, Washington, D. C.
Winton, A. L., Chemist, Connecticut Agricultural Experiment Station, New Haven, Conn.
Yocum, J. H., Chemist, United States Leather Company, 26 Ferry street, New York.

The secretary begged leave to make the following report of what has been accomplished in the matter of carrying out the order of the association to take steps to secure the coöperation of foreign chemists in testing methods of analysis:

New Brunswick, N. J., *December 20, 1893.*

Dear Sir: At the tenth annual convention of the Association of Official Agricultural Chemists resolutions were adopted authorizing the executive committee of the association to take such steps as they might find expedient to bring about a more general international coöperation in the study of methods of analysis. (See proceedings of the tenth annual convention, p. 121.) It seems desirable that the committee should take action on this matter at the earliest possible moment. Will you kindly inform me at once of the lines of action that you deem most expedient upon the part of the committee for the carrying out of this resolution? I ask this in order that, if possible, we may arrive at some concerted line of action without the trouble of a special meeting of the committee.

Very truly, yours,

E. B. Voorhees,
President.

Dr. H. W. Wiley,
Washington, D. C.

U. S. Department of Agriculture,
Division of Chemistry,
Washington, D. C., December 22, 1893.

Dear Sir: I suggest, in reply to your letter of December 20, that you, as chairman of the executive committee, address some of the leading members of the German union of agricultural chemists, Prof. Dehérain and other leaders of French agricultural chemistry, Prof. Voelcker and other leaders of English agricultural chemistry, telling them of the action taken by our association and suggesting that international coöperation be established for the purpose of unifying the methods employed for the examination of fertilizers and leading agricultural products throughout the world. I suggest that Prof. Maercker would be an appropriate person to correspond with in Germany, Prof. Dehérain in France, as I have indicated, and Prof. Voelcker in England. If France, Germany, England, and the United States agree upon methods, it will not be long before all other agricultural chemists of the world will be found cooperating. I think the most we can do between this and our next meeting is to get expressions from the gentlemen to whom I have referred of sympathy in the movement.

Let me suggest that you ask our reporters to send their circulars to a large number of French, German, and English chemists and get individual coöperation during the present year, as we did with potash last year. This will do much toward bringing about the desired unanimity of action.

I am, respectfully,

H. W. WILEY,
Chief of the Chemical Division.

Dr. E. B. VOORHEES,
President of the Association of Official Agricultural Chemists, New Brunswick, N. J.

NEW BRUNSWICK, N. J., *January 22, 1894.*

DEAR DR. WILEY: I inclose to you copies of circular letters, one to be sent to the various reporters and the other to foreign chemists, inviting their coöperation in our work, as per resolutions of the association. I am unfamiliar with such matters and I shall be pleased to have your criticisms and suggestions thereon. Will you kindly inform me whether the letter to the reporters covers the ground that you desire in reference to samples?

Very truly, yours,

E. B. VOORHEES.

Dr. H. W. WILEY,
Washington, D. C.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., January 27, 1894.

DEAR DR. VOORHEES: I think the circulars you have prepared are quite proper as they are. I have only made one or two unimportant changes. I return them for your approval. As you see, I have added your name as president of the association. If you find them now to meet with your approval, on their return I will have them printed and forwarded to the reporters, and also put myself in communication at once with prominent foreign chemists who would be likely to take part in the work. I think great good will come from this move.

Respectfully,

H. W. WILEY,
Secretary Association of Official Agricultural Chemists.

Dr. E. B. VOORHEES,
New Brunswick, N. J.

Circular letter sent by the president to the reporters:

DEAR SIR: In accordance with resolutions adopted at the last annual convention of the Association of Official Agricultural Chemists, in reference to coöperation in our work by foreign chemists, the executive committee of the association has requested the secretary, Dr. H. W. Wiley, to communicate with foreign chemists and chemical societies, asking their coöperation as set forth in the resolution adopted. In view of a possible acceptance of this invitation the executive committee desires that the various reporters prepare extra quantities of samples, and also directions, for the use of said foreign chemists, and that these be sent to Dr. Wiley, who will attend to their proper distribution.

EDWARD B. VOORHEES,
President Association of Official Agricultural Chemists.

Circular sent by the secretary to the reporters:

U. S. Department of Agriculture,
Division of Chemistry,
Washington, D. C., February 1, 1894.

Dear Sir: The Association of Official Agricultural Chemists has taken steps to secure a wider international coöperation in respect of methods of chemical analysis of substances pertaining to agriculture. The inclosed circular, approved by the executive committee of the association, will indicate the scope of the action desired. I beg you, therefore, in preparing samples for analysis by American chemists to provide a large number in excess of that needed in this country to supply any possible demand from European chemists.

I will send a circular at once to some of the prominent agricultural chemists in Europe, asking them to coöperate. If they consent to do so the samples which you have prepared will then be forwared to them. I think we may safely count on having ten foreign chemists coöperate in the work, and if, therefore, you will send me ten extra sets of samples I will have them on hand to meet any possible demand from Europe. The samples can be sent here by express, at our expense, and will be forwarded free of any cost to the association to any chemists in Europe making application therefor.

Respectfully,

H. W. Wiley,
Secretary Association of Official Agricultural Chemists.

By direction of the president of the association circulars were sent to foreign chemists connected with agricultural laboratories and experimental stations asking them to coöperate in the analytical work. The number of circulars sent was 270, to the following countries:

Austria-Hungary	34	Holland	4	Roumania	2
Belgium	9	Ireland	1	Russia	13
Denmark	3	Italy	18	Scotland	3
England	5	Japan	1	Spain	1
France	63	Norway	3	Sweden	24
Germany	7	Portugal	7	Switzerland	8

In most cases no reply was received to this circular letter and in all only about 25 responses were obtained. Of these the greater number regretted their inability to take part in the work. The following-named gentlemen, however, promised to coöperate, viz:

Name.	Locality.	Samples requested.
Dr. A. P. Aitken	Edinburgh, Scotland	Feeding stuffs.
Director, Den Kemiske Kontrolstation	Christiana, Norway	Phosphoric acid.
Dr. Fassbender	Kempen a Rh., Germany	Phosphoric acid, potash, nitrogen, feeding stuffs.
Dr. A. F. Holleman	Groningen, Holland	Phosphoric acid, nitrogen, feeding stuffs, sugar.
Prof. Dr. Roesler	Klosterneuburg, Austria	Wine.
Prof. Dr. J. Sebelien	Aas, Norway	Dairy products, feeding stuffs.
Dr. F. Strohmer	Wien, Austria	Phosphoric acid, feeding stuffs, sugar.
Dr. George Thoms	Riga, Russia	Phosphoric acid, potash, nitrogen, soils.
J. Cuthbert Welch	Reading, England	Wine.

To each of those promising coöperation the samples requested were sent, together with the full instructions prepared by the reporters on each subject, and a copy of Bulletin 38. When these samples were sent it was requested that the analyses be forwarded so as to be in the hands of the secretary of the association not later than the first of August. Only two of the chemists promising coöperation were able to send in their analyses by the time specified, viz, Dr. A. P. Aitken and Dr. Fassbender. Letters from the others have been received promising to send the results at a later date. A partial report from Dr. Thoms, of Riga, and a full report from Dr. Strohmer, of Vienna, were not received in time for transmission to the reporters.

In addition to this coöperative work the reporter on dairy products secured the assistance of Mr. H. Droop Richmond, of London, whose papers will be found in their appropriate places.

While the cöoperation asked for in foreign countries has not been very general, yet it certainly marks the beginning of a coöperative work which, if properly prosecuted, will result in the greatest benefit to chemists in all parts of the world, and it is suggested that further efforts be made to secure a wider coöperation of foreign chemists in our work.

Mr. Ross called attention to the fact that at the meeting in Chicago last year it was ordered that a committee be appointed to consider the recommendations made by the reporters in regard to methods of analysis. The presiding officer appointed as members of this committee the following gentlemen, to wit: B. W. Kilgore, H. J. Wheeler, A. M. Peter, H. J. Patterson, and G. L. Spencer. The presiding officer then called for the reading of the report on dairy products. The secretary read a letter from Mr. Farrington regretting his inability to be present on the first day of the meeting, and asking that his report be placed for the second or third day. Mr. Farrington not having yet arrived, it was moved to proceed with the regular order and to call for the dairy report at such time as would be convenient for the convention and the reporter when he arrived.

The presiding officer next called for the report on potash, which was presented by Mr. H. J. Wheeler.

REPORT ON POTASH.

By H. J. Wheeler.

In considering what line of work to take up, your reporter was convinced, in view of recent severe criticisms of one of our methods for potash determination, that a thorough test of the accuracy of present methods was demanded. If the methods of this association are charged with being faulty or inferior to others, it should be our immediate duty to investigate the question, looking to an improvement of present methods, the adoption of better ones, or a refutation of the charges in question. Reference is made in this connection more particularly to the statements by Breyer and Schweitzer* and to those embodied in a communication from the

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*Jour. Analyt. and Applied Chem., Vol. vi, No. 8, August, 1892.

German Kali Syndicate, entitled "A description and critique of various methods of potash analysis."

The general plan for the work on potash was formulated during December, 1893, and on the 26th of the same month a circular letter with a general outline of the plan was sent to over fifty chemists.

The final distribution of samples occurred on March 1, 1894. In all, 21 samples were sent out, 9 of which were intended for European chemists. After the complete elaboration of the above-mentioned plan and before having received a copy of the proceedings of the tenth annual convention, the attention of your reporter was called to a vote of the association referring the Stassfurt method to the reporter for this year, with directions "to make a recommendation thereon." Had this action of the association been known at the outstart, that method would have been embodied in the plan of investigation, but under the circumstances, considerable preliminary work having already been done, it was decided to make no further changes. In fact, the Stassfurt method had already been considered and was left out of the plan for the reason that it is only adapted to the analysis of potash salts and not to mixed fertilizers, solutions of which may contain phosphates, nitrates, and organic matter, impurities not met with in the analysis of commercial potash salts. To be sure, a method was used last year by the chemists of the potash syndicate in the analysis of similar material (sample No. 2), but it was evidently adopted to meet the case in hand and could not be considered as a Stassfurt method. The choice of a test involving such impurities as are met with in the analysis of our ordinary mixed fertilizers, instead of one with those contained in commercial potash salts, was made, for the reason that the greater portion of the practical work of this association is in connection with the analysis of mixed goods and not of potash salts, and it seemed more important to devote our first efforts to a careful test under those conditions with which we must continually deal and to undertake on some later occasion an investigation of methods for conducting those tests which are only made occasionally. The almost universal complaint of lack of time for the work showed plainly the inadvisability of taking up more than one question at a time. The general scheme of the investigation is outlined in the following circular of instructions which accompanied each set of samples:

CIRCULAR.

KINGSTON, R. I., *March 1, 1894.*

DEAR SIR: I send you to-day by express, for the work on potash for the Association of Official Agricultural Chemists, two bottles of materials. One contains a c. p. sample of muriate of potash and the other a solution of "impurities," containing sulphuric acid, phosphoric acid, nitric acid, lime, magnesia, soda, and organic matter in form of cane sugar. In the preparation of the "impurities" only so-called c. p. reagents were employed, and each 25 cc. contains about twice the amount of the above-mentioned impurities found in the watery extract from one-half gram of a sample representing the average of our mixed fertilizers.

PREPARATION OF POTASH SOLUTION.

The potash solution is to be made so that each liter shall contain 4 grams of the c. p. KCl.

A. Tests by Alternate method (Proceedings Tenth An. Convention, A. O. A. C., p. 179):

1. Employ 25 cc. of the KCl solution and 25 cc. of the impurities.
2. Employ 25 cc. of the impurities.
3. Employ distilled water, using the same amount of $BaCl_2$ and $Ba(OH)_2$ as in 1 and 2 and as much $(NH_4)_2\ CO_3$ as necessary.
4. Evaporate 25 cc. of the KCl solution with $PtCl_4$ and finish the test as in the Alternate method.
5. Evaporate the same amount of $PtCl_4$ as employed in the above tests, without KCl solution, and finish the test as in the Alternate method.

B. Tests by Lindo-Gladding method (Proceedings Tenth An. Convention, A. O. A. C., p. 183):

1. Take 150 cc. of the KCl solution and 150 cc. of the impurities and evaporate the mixture sufficiently to allow of the addition of the necessary ammonia and ammonium oxalate (which are to be added to the hot solution), so that the whole can be made up to 300 cc. after cooling. Filter through a dry filter and employ 50 cc. of this solution in each case for the completion of each test.
2. Take 150 cc. of impurities and enough distilled water so that the heated solution will be of about the same volume as in 1, add ammonia and ammonium oxalate and proceed as in 1.
3. Add to the ammonia and ammonium oxalate sufficient distilled water to equal 300 cc., then take 50 cc. of this solution and proceed as in 1 and 2.
4. Evaporate 25 cc. of the KCl solution with $PtCl_4$, and finish the test as in the Lindo-Gladding method.
5. Evaporate the same amount of $PtCl_4$ as employed in the above tests, without KCl solution, and finish the test as in the Lindo-Gladding method.

MOISTURE TESTS.

Take 5 grams of the c. p. KCl and dry at 130° C. to constant weight.

DIRECTIONS.

At the outstart make sure that you have enough of each of the reagents on hand so that the same lot may serve for all of the tests.

In the case of each method, use the same amounts of reagents for all of the tests given under that method (excepting test 3 under A, as there specified), and to this end make test 1 under each method at the outstart, to determine the amount of reagents required.

For the sake of perfect uniformity in the work, please follow strictly the methods as laid down by the Association of Official Agricultural Chemists, taking especial care when evaporating with $PtCl_4$ to remove from the water bath just as soon as the point is reached at which the material will entirely crystallize upon cooling. Do not allow the solution to come to dryness on the water bath. If not convenient to treat with alcohol at once, place in a desiccator to prevent absorption of moisture. In regard to the treatment with 80 per cent alcohol, we would advise the following method: When cool, add about 30 cc. of alcohol, pulverize the salt and let stand about twenty minutes with frequent stirring, decant on the filter, wash by decantation with successive portions of about 10 cc. of alcohol, allowing each portion to remain in contact with the salt for about five minutes, stirring frequently. Complete the washing as directed in the proceedings of the Association of Official Agricultural Chemists. Do not collect two or more precipitates upon the same filter, but prepare a new Gooch filter for each precipitate, washing with the same solutions as used subsequently.

If, upon completion of all the work outlined above, you still have sufficient material and time, it is suggested that you make additional tests by both methods, in which the evaporation with $PtCl_4$ shall be continued one-half hour after perfect dryness is reached, for the purpose of ascertaining the relative danger of reduction of $PtCl_4$ in the Lindo-Gladding and Alternate methods. This is proposed for the reason that greater difficulty is experienced in the Alternate method in removing all traces of organic matter and that for this reason there may be greater danger of reduction than with the Lindo-Gladding method. If such proves to be the case it would serve to emphasize the necessity for extreme care in evaporation when using the Alternate method.

In reporting, please give the weight of the precipitate in each reliable test, instead of averages or percentages of K_2O. Please send in your report as early as July 1, and earlier if possible.

If you find it necessary, for any reason, to deviate in any way from these directions or those laid down by the Association of Official Agricultural Chemists (above referred to) please state in full all details relating to the same.

In the potassium chlorid* sent out no impurities could be detected other than a faint trace of iron and a little soda. In an attempt to further purify it a portion was subjected to several recrystallizations, though, as shown by analysis, without any apparent advantage.

As a basis for the preparation of impurities a mixture was made of about equal quantities of fifty different brands of complete commercial fertilizers, and a watery solution of 10 grams of this mixture, prepared as for potash determinations, was submitted to analysis with the following result:

Amount of soluble constituents in 1 gram of the above mixture.

	Grams.
Calcium oxid (CaO)	.0490
Magnesium oxid (MgO)	.0075
Sodium oxid (Na_2O)	.0301
Potassium oxid (K_2O)	.0356
Phosphoric acid (P_2O_5)	.0556
Sulphuric acid (SO_3)	.0378
Chlorin	.0330

Silica, iron, and alumina were not present.

The solution of impurities was prepared in such a way that the amount contained in each 25 cc. was practically equivalent to that in the watery extract from 1 gram of the above fertilizer and the mixture was prepared from the following so-called c. p. chemicals:

Amounts of chemicals employed for 12 liters of "impurities."

13.82 grams H_2SO_4 (1.84 specific gravity).
83.33 grams $Na_2H\ PO_4+12$ aq.
11.47 grams HPO_3.
22.08 grams $MgSO_4+7$ aq.
68.83 grams $Ca(NO_3)_2$.
10.00 grams cane sugar.

Cane sugar was added to furnish organic matter, so that the conditions would more nearly resemble those of ordinary practice.

The following tabular statements show the results obtained by the different analysts:

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* This salt was chosen for the reason that its solution could be evaporated directly with $PtCl_4$, and as the "impurities" contained considerable sulphuric acid, all the difficulties encountered in the analysis of the sulphate would be met with to some extent.

A.—*Results of potash work for 1894 as received.*

[Given in milligrams of K_2PtCl_6.]

Source of analysis.	Water.	1 KCl and impurities.		2 Blank impurities and reagents.		3 Blank reagents.		4 Direct evap. KCl and K_2PtCl_6.		5 Blank $PtCl_4$.	
		Alt.	L.-G.	Alt.	L.-G.	Alt.	L.-G.	Alt.	L.-G.	Alt.	L.-G.
	P. ct.										
Colorado College, William P. Headden	(*)	325.9	325.1	......	1.4	......	......	327.1	......	......	......
	......	324.7	324.5	......	1.9	......	......	326.9	......	......	......
	......	326.0	325.5	......	......	......	......	......	......	......	......
	......	......	325.2	......	......	......	......	......	......	......	......
	......	......	325.9	......	......	......	......	......	......	......	......
Illinois Station, W. A. Powers	0.093	328.0	324.5	6.5	4.2	2.4	1.2	327.6	330.2	.3	.2
	.093	329.5	324.6	6.0	5.3	2.3	1.6	329.6	331.0	.4	.4
	......	328.7	324.7	......	......	......	......	......	......	......	......
Maryland College, F. P. Veitch	.16	333.0	327.4	12.7	6.8	12.2	5.3	327.0	326.9	6.0	6.0
Massachusetts Station, J. B. Lindsey	(*)	325.0	318.0	8.0	3.9	7.1	1.0	329.5	328.5	.0	.0
	......	324.6	319.8	7.0	3.5	5.2	1.8	328.0	329.0	......	......
	......	324.8	319.8	8.5	2.0	......	3.5	328.0	327.8	......	......
	......	......	......	......	......	......	......	......	326.0	......	......
Michigan Station, W. L. Rossman	.06	327.3	324.4	1.5	1.3	1.0	.5	324.5	324.8	.0	.0
	......	325.0	324.3	1.0	1.0	1.2	1.0	324.3	324.2	.5	.0
	......	......	324.0	......	......	......	......	324.5	324.7	......	......
	......	......	......	......	......	......	......	323.7	324.5	......	......
New Jersey Station, J. P. Street	(†)	326.1	320.6	3.0	3.0	2.9	.9	321.4	320.4	.8	.8
	......	327.1	320.7	3.0	3.5	3.0	1.1	322.4	321.4	.9	.9
	......	......	322.8	......	......	......	......	322.5	......	......	......
Rhode Island Station, B. L. Hartwell	.077	326.2	330.6	5.4	10.4	1.4	1.2	324.6	322.6	.3	.4
	......	......	......	6.0	......	......	......	......	323.1	.3	.3
	......	326.1	330.2	6.8	10 2	1.5	1.4	324.4	323.8	......	......
	......	......	......	7.2	......	......	......	......	......	......	......
U. S. Department of Agriculture, K. P. McElroy	.06	329 3	329.7	1.7	.4	.0	.0	325.6	326.1	.0	.0
	.08	326.5	329.0	1.8	.4	......	......	326.0	326.9	......	......
West Virginia Station, R. de Roode	(*)	325.7	326.0	2.6	3.9	4.8	.5	326.3	326.0	.6	.4

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* Results given on a dry basis.

† Drying three hours caused no loss in weight.

B.—*Average results of potash work for 1894 calculated to water-free potassium chlorid.*

[Given in milligrams of K_2PtCl_6.]

Source of analysis.	1 KCl and impurities.		2 Blank impurities and reagents.		3 Blank reagents.		4 Direct evaporation. KCl and $PtCl_4$.		5 Blank $PtCl_4$.	
	Alt.	L.-G.	Alt.	L.-G.	Alt.	L.-G.	Alt.	L.-G.	Alt.	L.-G.
Colorado College, Wm. P. Headden	325.5	325.2	......	1.7	......	......	327	......	......	......
Illinois Station, W. A. Powers	329	324.9	6.3	4.8	2.4	1.4	328.8	330.9	0.4	0.3
Maryland College, F. P. Veitch	333.5	327.9	12.7	6.8	12.2	5.3	327.5	327.4	6.0	6.0
Massachusetts Station, J. B. Lindsey	324.8	319.2	7.8	3.1	6.2	2.1	328.5	327.8	.0	.0
Michigan Station, W. L. Rossman	326.3	324.5	1.3	1.2	1.1	.8	324.4	324.8	.3	.0
New Jersey Station, J. P. Street	326.6	321.4	3.0	3.3	3.0	1.0	322.1	320.9	.9	.9
Rhode Island Station, B. L. Hartwell	326.4	330.7	6.4	10.3	1.5	1.3	324.8	323.4	.3	.4
U. S. Department of Agriculture, K. P. McElroy	328.1	329.6	1.8	.4	.0	.0	326.0	326.7	.0	.0
West Virginia Station, R. de Roode	325.7	326.0	2.6	3.9	4.8	.5	326.3	326.0	.6	.4

C.—*Deductions from the above table.*

	1–2 KCl and imp. (1), minus blank for impurities and reagents.				4–5 Direct evap. KCl and $PtCl_4$ (4), minus blank for $PtCl_4$ (5).			
	K_2PtCl_6 (mg.).		KCl (per cent).		K_2PtCl_6 (mg.).		KCl (per cent).	
	Alt.	L.-G.	Alt.	L.-G.	Alt.	L.-G.	Alt.	L.-G.
Colorado College, Wm. P. Headden	....	323.5	....	98.86	....	....	....	....
Illinois Station, W. A. Powers	322.7	320.1	98.63	97.82	328.4	* 330.6	100.36	* 101.03
Maryland College, F. P. Veitch	320.8	321.1	98.04	98.13	321.5	321.4	98.25	98.22
Massachusetts Station, J. B. Lindsey	* 317.0	* 316.1	* 96.88	* 96.60	328.5	327.8	100.39	100.18
Michigan Station, W. L. Rossman	325.0	323.3	99.32	98.80	324.1	324.8	99.04	99.26
New Jersey Station, J. P. Street	323.6	318.1	98.89	97.21	321.2	320.0	98.16	97.79
Rhode Island Station, B. L. Hartwell	320.0	320.4	97.79	97.91	324.5	323.0	99.17	98.71
U. S. Department of Agriculture, K. P. McElroy	* 326.3	* 329.2	* 99.72	*100.60	326.0	326.7	99.63	99.84
West Virginia Station, R. de Roode	323.1	322.1	98.74	98.43	325.7	325.6	99.53	99.50
Maximum	326.3	329.2	99.72	100.60	328.5	330.6	100.39	101.03
Minimum	317.0	316.1	96.88	96.60	321.2	320.0	98.16	97.79
Average †	322.5	321.2	98.56	98.16	324.9	324.2	99.29	99.08

* Not included in calculating the "average."

† Does not include results marked (*).

In drawing up the above averages the individual results secured by the different analysts were reduced to a dry basis for the KCl, using in each case the percentage of moisture as found. The method finally pursued by two of the analysts in the determination of moisture, viz, that of evaporating and heating an aliquot part of the KCl solution is superior to that suggested by your reporter, for the reason that it at the same time makes a correction for possible errors in pipettes or burettes; yet it is to be hoped that such care has been taken by the various analysts that no appreciable errors have resulted from this cause. All extraordinarily high or low results were omitted in calculating the averages. Two analysts reported that their $PtCl_4$ gave no blank after evaporation and treatment with alcohol, and one reported no blank from any of his reagents. The experience of the majority of the analysts shows that even in cases where, from a casual observation, no insoluble residue was observed, a slight blank was nevertheless obtained, indicating the necessity for the filtration of blanks in all cases.

In preparing the general table of averages (Table B) it becomes necessary to average separately the determinations made by each analyst, for the reason that the amounts of blank due to the difference in the quality of $PtCl_4$ and other reagents varied in each instance. Table C is derived directly from Table B, and shows the final amounts of K_2PtCl_6 obtained after the deduction of the respective blanks. In making up the averages from Table C certain exceptional results were omitted.

A careful study of the results shows that the blanks obtained by direct evaporation of $PtCl_4$ gave, as would be expected, practically identical results by both methods of washing. It appears also that the amount of potash in the "impurities" must, if present, have been small.

There is apparently no reasonable ground why the amount of K_2PtCl_6, obtained by direct evaporation and washing with 80 per cent alcohol and ether, should not, after deduction of the blank found by direct evaporation of the $PtCl_4$, represent most nearly the actual amount of potash, and it should therefore be taken as a fair standard for a comparison of the results by the two methods, when the potash was determined in the presence of impurities. Even the results obtained by such direct evaporation may be slightly low, owing to the well-known slight solubility of K_2PtCl_6 in 80 per cent alcohol, and furthermore owing to the possibility of slight traces of HCl remaining behind, which would still further increase its solubility. Accepting, however, the average of the results in the third column of Table C as a standard, it will be seen that those obtained by direct evaporation and subsequent washing with the NH_4Cl wash solution employed in the Lindo-Gladding method are extremely close, a fact which indicates that in analyses in which no more K_2PtCl_6 is produced than in these tests there is, when washed in the customary way and with the regulation quantity of the solution, no considerable loss by double decomposition on the filter. One or two observations * are on record showing that such double decomposition does result; and your reporter, to further test this question, asked the assistance of Mr. Voorhees in making such a test, and the execution of the work was intrusted to his assistant, Mr. Street. In making this test it was requested that a large quantity of K_2PtCl_6 which had been tested for NH_3 should be subjected for some days to the regular NH_4Cl wash as employed in the Lindo-Gladding method. Admitting that such double decomposition is possible, it seemed necessary, in settling definitely whether such is the case or not, to exaggerate the conditions of treatment so far as concerned the quantities of K_2PtCl_6 and the time allowed for double decomposition to take place. If double decomposition takes place to any extent the K_2PtCl_6 which is submitted to the action of the wash ought to show the presence of an amount of ammonia far beyond that arising from any possible analytical error, but working under ordinary conditions, even though such decomposition resulted, it might still be a question whether the variations in the data secured were not wholly within the limit of analytical error.

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* Breyer and Schweitzer, Jour. Analyt. and Applied Chem., Vol. VI, No. 8, Aug. 1892. Also N. Robinson, Jour. Am. Chem. Soc., Vol. XVI, No. 6, June, 1890, p 364.

The following are the results as reported by Mr. Street:

Solubility of K_2PtCl_6 in NH_4Cl wash.

"The salt was allowed to stand in the NH_4Cl wash, with frequent stirring, for six days.

Weight of salt originally.	Weight of salt after 6 days.	Loss.
Grams.	*Grams.*	*Grams.*
1.5678	1.4934	0.0744
1.5657	1.4840	0.0817

Test for ammonia in K_2PtCl_6 salt.

"Two samples were taken which had been washed with alcohol only:

1.5688 grams K_2PtCl_6 gave	0.000156 grams N.
1.5681 grams K_2PtCl_6 gave	0.000312 grams N.

"The two samples which stood in NH_4Cl for six days, after thorough washing with alcohol, were tested for ammonia:

1.4934 grams K_2PtCl_6 (?) gave	0.028392 grams N.
1.4840 grams K_2PtCl_6 gave	0.025896 grams N.
A blank determination gave	0.000156 grams N."

These results further confirm the opinion that double decomposition of K_2PtCl_6 actually results when it is brought in contact with the NH_4Cl wash. To be sure, these results were secured under extraordinary conditions as to time of treatment, etc., yet they do indicate that there may be a source of error, though perhaps small, from this cause. As seen in Table C, the average amount of error resulting under the conditions of ordinary practice was but 0.7 of a milligram for the Lindo-Gladding method gave, after deducting the blank for $PtCl_4$, 324.2 milligrams, and the alternate 324.9 milligrams.

Comparing the averages obtained from Table C, it will be seen that the amount of K_2PtCl_6 recovered, when determined in presence of the "impurities," is less in case of both methods than that obtained by direct evaporation; and, furthermore, it will be observed that the average of the results secured by the alternate method are 1.3 milligram nearer that obtained by direct evaporation, than is the average secured by the Lindo-Gladding method. It appears possible that the lower results in both cases may be due to some extent to the occlusion of potash salts, which is liable to be unequally great by the two methods. In the case of the alternate method there is also liability to loss by insufficient washing and by volatilization of KCl in driving off the ammonia salts. Messrs. De Roode and Lindsey also called attention to the danger from loss by spattering in the evaporation with H_2SO_4 in the Lindo-Gladding method and the latter method also has, as shown, a tendency to slightly lower results probably due to double decomposition, though, as heretofore stated, this loss appears to be slight when working under normal conditions.

In an article by Dr. Robinson, of Florida (Jour. of the Am. Chem. Soc., Vol. XVI, No. 6, June, 1894, p. 364), attention has been called to the occlusion of potash salts even in acid solutions and the danger of loss arising therefrom in various methods of analysis, a point which he was not aware had been heretofore studied. It appears, however, to be well known, for West (Zeit. für analyt. Chemie 20, 1881), as far back as 1881 recognized and called attention to it. Crookes, in his "Select methods in chemical analysis" (London), also calls attention to this fact, and in the proceedings of this association for 1885 it is also referred to as "well known," and the statement was made by the committee on potash, that for this reason "the addition of barium chlorid and of barium hydrate and ammonium carbonate should be made as indicated in the report of the committee, drop by drop, and in as slight excess as possible."

In the work by Dr. Robinson and in that by Breyer and Schweitzer, which involved the recovery of occluded potash as well as that removed by the NH_4Cl and alcohol washings, your reporter has found no statement to show that the reagents therein involved had previously been tested for potash, and provided such precautions were omitted, the amount of potash claimed by them to have been lost by occlusion, double decomposition, etc., may have been overstated, for, as will be seen from the foregoing tables, the blanks with reagents, after deducting that for the $PtCl_4$, amounted in some cases to several milligrams for each determination. It would, therefore, not seem surprising if the 7 grams of $Ba(OH)_2$ and other reagents involved in Breyer and Schweitzer's investigation may have been responsible for a part of the potash claimed to have been recovered from the washings in question.

Two analysts in particular (Lindsey and de Roode) found great difficulty in securing even fairly concordant results with the Lindo-Gladding method, when it came to a determination of the potash in the presence of impurities, a point which militates considerably against that method as compared with the Alternate. Any method is more or less unsatisfactory which, when carefully worked, will not give fairly concordant results in the hands of the same analyst.

On the other hand, unless great care is exercised in evaporating with $PtCl_4$ there is (owing to organic matter liable to be present), presumably, greater danger from reduction of $PtCl_4$ in the alternate than in the Lindo-Gladding method.

Only one result was, however, reported in this connection, and consequently it throws no conclusive light on this question.

The following report was received from one German chemist, but as the results have no bearing on the comparison of the two official methods which was the chief object in view, they are included without further comment:

RESULTS ON OFFICIAL SAMPLES AND DESCRIPTIONS OF METHODS EMPLOYED.

By Dr. Fassbender.

POTASH DETERMINATION.

Four grams of substance were dissolved in a liter of water and a 25 cc. portion acidulated with hydrochloric acid. Platinum chlorid was added and the mixture evaporated till the smell of hydrochloric acid had disappeared. The excess of platinum chlorid was then washed out with 90 per cent alcohol, washing being continued till the filtrate appeared colorless. The residue of potassium platinum chlorid was dissolved in hot water and passed through a filter into a beaker. The solution was acidulated with hydrochloride acid, formate of sodium added, and the solution warmed till the platinum was all separated. The supernatant fluid appeared clear and colorless. The platinum was brought on a filter, washed with water containing hydrochloric acid, and finally ignited in a platinum crucible.

	Platinum.	K_2O.
	Gram.	*Gram.*
25 cc., or 0.1 gram	0.1325 0.1323	0.0631 0.0630

Impure solution.—Twenty five cubic centimeters of the solution just described were mixed with 25 cc. "impurities" and the whole evaporated down to dryness in a platinum dish. The residue was faintly ignited, moistened with concentrated hydrochloric acid, evaporated down, dried, and once more ignited. The residue was dissolved in hot water, the solution feebly acidified, barium chlorid added till all sulphuric acid was precipitated and then baryta water to alkalinity. Ammonium carbonate was then added till no more precipitate formed, the liquid filtered, washing the residue well, the filtrate evaporated down in a platinum dish, and the residue dried and ignited to expel the ammonia salts. The residue was taken up with hot water, filtered, and in the filtrate the potash estimated as previously described.

	Platinum.	K_2O.
	Gram.	*Gram.*
25 cc., or 0.1 gram	0.1330	0.0633
	0.1319	0.0628

No further papers being presented, discussion of the report was declared to be in order.

Mr. PAYNE spoke of discrepancies in the determinations of potash. Last year 153 determinations were made and the discrepancies in results were probably due to lack of mechanical mixing. It is important that the sample be in a finely mechanical condition. Where the variations are as much as 1 per cent they must be due largely to chemists instead of methods. It is always important to use plenty of the platinic chlorid.

Mr. WHEELER called attention to the difficulty which was encountered in carrying on strictly scientific work, in the nature of research, experienced by those whose time is wholly occupied with routine work in the analysis of fertilizers. It is important that those who are doing the official work for the association should be qualified in every respect for the solution of the problems which they undertake.

Mr. LINDSEY said he thought some of the irregularities in the work on potash should be ascribed to the impurities in the reagents employed. He himself used Trommsdorf's chemicals, but had found considerable irregularities arising from the impurities which they contain.

Mr. WHEELER called attention to the fact that he had omitted from his report data obtained by Dr. Fassbender, of Germany, but that they would be published as a part of the proceedings of the association. Dr. Fassbender did not use the methods employed by the association, but the results which he obtained were almost identical with those given in his report. Dr. Fassbender reduced the potash salt at the end of the operation, and determined the amount of potash by the weight of metallic platinum obtained.

Mr. HUSTON spoke of the behavior of superphosphates containing much organic matter, such as tobacco stems. In many cases where the Lindo-Gladding method was used, he had found that the double platinum salt obtained contained notable quantities of phosphorus. The difficulty can be removed by adding a solution of calcium chlorid before the addition of ammonia and ammonium oxalate or carbonate. He stated that he had considerable work in progress to show whether there was any objection to the use of the calcium chlorid. He made the preliminary statement for the purpose of calling attention to a common source of error in the Lindo-Gladding method.

Mr. WHEELER said that in the preparation of the materials containing potash for final precipitation, it was the barium salts employed which specially carried down the potash mechanically, and that the loss of potash should be looked for in the precipitated barium salts.

Mr. HUSTON remarked that was the reason he preferred to use calcium chlorid instead of barium chlorid in these precipitations.

Mr. WHEELER said that the reporter had a few recommendations in regard to changes in the methods of analysis, and they looked more to uniformity in the various parts of the work. For instance, where it was directed in one part to boil ten minutes and in another thirty minutes, he thought a uniform time should be used for both. He also suggested a further comparison of methods in cases of samples containing large quantities of sulphates. The various recommendations of the reporter were referred to the committee appointed to consider these matters.

Mr. FREAR asked if any member might present recommendations which the reporter had made, and which the committee did not concur in, for further action of the association. The presiding officer replied that any member had this privilege, but that recommendations of the reporter which were not concurred in by the committee were required to be adopted by a two-thirds vote of the association.

Mr. ROSS suggested that in the case of the analysis of potash fertilizers, something more explicit should be stated in regard to the treatment of those samples containing organic matters.

Mr. DE ROODE remarked that only the potash soluble in water, according to the laws of his State, could be reported in the sample. In cases of potash samples containing this material in organic form not soluble in water, he would not be allowed to incinerate the sample before analysis and thus obtain practically the whole of the potash.

Mr. MCDONNELL fully concurred in the recommendations of the reporter, that the methods of treatment in the analytical processes should be as uniform as possible, and, therefore, where there were two methods recommended for washing the precipitate, viz, one with alcohol and the other alcohol and ether, he thought it would be well to change them so as to make them both alcohol and ether or both alcohol only.

Mr. WHEELER further suggested that in the methods of analysis errors due to typographical mistakes alone should be corrected either by the reporter or by the editor.

THE SECRETARY in this connection stated that, while he was allowed great freedom in the editing of the body of the report, he did not feel at liberty to make any changes in the methods of analysis, even of a typographical nature, after they had finally passed out of the hands of the reporter. The association had always held the reporters responsible for the correct form of the methods which they delivered to the secretary for publication; nevertheless, in the case of glaring typographical errors, there would probably be no objection to their correction by the editor during the process of printing.

The presiding officer suggested that all these matters be taken into consideration by the committee and acted upon.

The secretary pointed out the fact that the time at the disposal of the association was limited, and that the committee to consider recommendations of the reporters should act as speedily as possible, and that it was advisable for them to meet during the evening if it could be arranged.

All matters relating to the changes in the methods of potash analysis having been referred to the committee, there was no further discussion of Mr. Wheeler's report.

The presiding officer then called for the report on soils and ash analysis.

Mr. Peter then read his report. During the reading of Mr. Peter's report, the committee which had been appointed to wait on the assisant secretary appeared and stated that the duty assigned to it had been performed, and that the assistant secretary had consented to visit the association at 2 o'clock p. m. The report was accepted and the committee discharged.

At 12:30, Mr. Peter not having yet finished his report, a recess for luncheon was taken in harmony with an order of the committee on rules. After luncheon Mr. Peter continued his report, and the recommendations he made for changes in the methods of analysis were referred to the committee.

REPORT ON SOIL AND ASH.

By Alfred M. Peter.

At the last meeting of this association the reporter on soils and ash presented as his report a detailed description of the method of soil analysis practiced by Drs. Hilgard, Peter, and others, and this report was referred by the association, without discussion, to the reporter for the present year. The reporter of last year presented no results of coöperative work by members of the association, nor did he treat in any but a general way of the important recommendation of the previous reporter in regard to reducing the time of digestion, a recommendation that had been formally referred to the reporter of last year by a vote of this association at its meeting in 1892. In this state of affairs it was evident to your reporter that he was already charged with the investigation of more questions than would be enough to fully occupy him and his coworkers without bringing in any new matter. He therefore concluded to send out early in the year a circular to the chemists of the country asking suggestions as to which of a number of questions, bearing mainly on the discussion of the Hilgard and "provisional" methods, it would be best to take up, and asking coöperation. About 200 copies of this circular were sent out, and replies were received from 55 chemists, 30 of whom signified their willingness to undertake the analysis of material to be sent out by the reporter. After considering the suggestions made by many of these correspondents, your reporter decided to direct the work toward a comparison of results obtained upon identical soil samples by the three methods of digestion under consideration, and in addition suggested a trial of the method of digestion proposed by Mr. Harry Snyder, of Minnesota; also, a test of the action of HCl on glass and porcelain vessels; a test of the method of Huston & McBryde for humus; a determination of moisture by drying in the water oven, and mechanical analysis by Hilgard's or Osborne's methods as subjects that might be taken up where the time and inclination of the chemist would permit.

Two samples of soil and one of ash were first sent out, and later another sample of ash was distributed and one of soil, to a few chemists. The soils were prepared by sifting first through a 20-mesh sieve, being approximately a 1-millimeter mesh, thoroughly mixed and then sifted through a 40-mesh sieve, approximately one-half millimeter. The material removed by the 40-mesh sieve was mainly iron gravel, and amounted to 2 per cent in No. 2 and 4.33 per cent in No. 3. The fine earth was again carefully mixed and put into a tight vessel, where it remained several days to allow the moisture content to become perfectly uniform. It was then bottled quickly, corked, and sealed, and was ready for distribution.

The ash samples were prepared by burning the material in a large iron stove, avoiding intense heating as much as possible. The fused portions were sifted out on the 20-mesh sieve, the fine part being rubbed up in a mortar with a rubber pestle and passed successively through a 40 and 60 mesh sieve, rejecting each time hard, fused particles. The ash was then heated to low redness for a short time in iron pans, sifted again and well stirred with a magnet to remove any particles of iron, and bottled in the same way as the soils, after having remained some time in a close vessel. The samples distributed were:—

No. 1. Ash of clover hay.

No. 2. Virgin soil taken to the depth of about 6 inches from a woodland bluegrass pasture adjoining the experimental field of the Kentucky station. Present forest growth, ash, elm, honey locust, coffee bean, hickory, black walnut, and sugar maple.

No. 3. Cultivated soil from an unfertilized plot in the experimental field of the Kentucky station, taken also to the depth of about 6 inches. This soil has been in cultivation many years, but is believed never to have received any fertilizer.

No. 4. Ash of corn "stover."

No. 5. A Kentucky soil with moderate percentage of P_2O_5. This was sent to only a few of the chemists for the special purpose of testing the Goss method for P_2O_5 determination.

Thirty-one sets of samples were distributed, including one to Europe and one to Canada. The following instructions were sent with the samples, and later a description of the method of Arthur Goss for P_2O_5 was sent out to all who had received samples:

"It is not expected that each chemist will undertake the whole of the work laid out below, but it is desired that preference be given the subjects in the order of their numbers. Where there is not time or opportunity for making a complete analysis of the soil samples, determine at least phosphoric acid, potash, insoluble matters and nitrogen. This partial analysis is all that is required in No. 3.

"The samples are ready for analysis and need only to be mixed in the bottles with a spatula or by rolling before weighing out.

"THE SOILS.*

"1. Analyze samples 2 and 3 by Hilgard's method (Bull. 38, p. 76) and by the Provisional method (p. 200), following the directions in each case as closely as possible. The insoluble residue from the nitric acid digestion by the Hilgard method, p. 80 (12), should be tested for phosphoric acid, either after decomposition by alkaline carbonates or digestion with strong hydrochloric acid.

"2. Try digestion for two hours, as recommended by the reporter for 1892 (Bull. 35, p. 107), and analysis by the Provisional method.

"3. Try digestion in a Snyder flask (Jour. An. and App. Chem., VII, 6) or in an Erlenmeyer flask covered with a watch glass or small funnel.

"4. Determine moisture by drying two grams to constant weight in a water oven.

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*References are to bulletins of the Division of Chemistry, U. S. Department of Agriculture, when not otherwise stated.

"5. Digest 25 cc. hydrochloric acid, 1.115 sp. gr., for five days in a porcelain beaker on the water bath, evaporate in a platinum dish and determine the weight of the residue after heating barely to redness. Repeat the experiment with a Bohemian glass beaker and also determine the residue left by evaporating 25 cc. of the same HCl directly in a platinum dish.

"6. Determine humus by the method of Huston & McBride (Bull. 38, p. 84; Indiana Bull. 46) and by the Grandeau method (Bull. 38, p. 80).

"7. Make mechanical analysis by Hilgard's method (Bull. 38, p. 62) and that of Osborne (Conn. State Rep., 1886, p. 144).

"THE ASH.

"Sample No. 1 is to be analyzed by the provisional method (Bull. 38, p. 206), not omitting to determine carbon and sand. The sample being small, it is suggested to determine carbon dioxid and chlorin in the portion used for moisture and carbon (1) and (2). As the precipitate of ferric phosphate obtained in (9), p. 207, is usually very hard to filter, it is suggested to add enough c. p. ferric chlorid solution to produce a red precipitate that is much easier to filter and wash, thus obtaining a clear filtrate for the Mn, Ca, and Mg determinations. Where the quantity of the ash is limited, this precipitate may be dissolved in nitric acid and the solution used for the phosphoric acid determination. Of course iron will have to be determined in another portion, or, if ash is scarce, the ammonium hydrate precipitate from (13) might be used for this purpose.

"The following method for C, SiO_2, and sand is proposed for trial: Proceed as in (3), p. 206, but filter on a weighed Gooch filter having the felt covered with a perforated disk of platinum (Jour. Am. Chem. Soc., xv, 12, 710). After washing thoroughy, remove as much of the residue as possible from the Gooch and treat it with boiling Na_2CO_3 solution as usual, using the same Gooch for the filtration. After thorough washing, dry at 110°, and weigh C and sand; then burn and weigh sand. The Na_2CO_3 filtrate may be acidified with HCl and evaporated for SiO_2.

"The reporter will also be glad to have the coöperative work called for above extended to other samples than those sent out by himself, whenever the operator can do so conveniently.

"Please calculate your results upon the samples as received, and report them as early as possible, making it clear in your report exactly how the analyses were made."

No reports of mechanical analyses were received; 18 chemists reported chemical analyses, the results of which are given in the following tables.

It will be seen that the amount of work done has been large and its character arduous, and your reporter desires here to express his thanks to all who have joined in the work, for their hearty coöperation. The mass of figures is large, and, although the results are often discordant, it is believed that they teach important lessons and will be a mine of material from which the reporter for next year may draw much that is of value. The present writer has not had time for a discussion of the results and can only present them in a compact form, touching only upon the main points proposed for investigation at the outset.

A table of averages is added for the purpose of comparing the Hilgard and Provisional methods. In making these averages all results are used, except only a few that seemed to differ too much from the next higher or lower, and the range between the highest and lowest is given as per cent of the average. This method is followed solely for the purpose of comparing the range of variation in the results by the two methods in the hands of different observers and not to arrive at the probable amounts of the constituents actually present. With the latter object in view it would have been necessary to reject many more figures, and the averages would in nearly every instance have been perceptibly different from those given. The results, taken as they come, are too discordant to justify taking the average of all as representing the most probable figure.

Table 1.

ASH SAMPLE No. 1.

[Provisional method.]

	R. J. Davidson and T. L. Watson, Virginia.	L. M. Bloomfield, Ohio.	R. J. de Roode, West Virginia.	F. L. Arnold, Massachusetts.	W. D. Bigelow, Department of Agriculture.	John Thompson, Minnesota.	Chas. Ryan, Colorado.	W. L. Rossman, Michigan.	Average.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Soluble SiO_2	1.18	7.385	3.43	....	....	....	4.14	....	....
Insoluble SiO_2	10.77	4.785	8.32	....	....	....	7.39	....	....
Total SiO_2	11.95	12.170	11.75	*14.36*	[1]*13.17*	11.81	11.53	12.076	11.891
K_2O	25.68	*29.250*	25.60	26.26	25.55	*24.21*	....	25.400	25.698
Na_2O	1.51	....	.32	....	.06	1.33	....	1.602	....
CaO	27.52	*22.345*	27.28	27.04	....	26.27	26.07	27.500	26.947
MgO	7.75	7.143	7.32	....	....	7.43	7.86	....	7.500
Mn_3O^4	....	.840	....	....	....	....	.09	....	....
Fe_2O_3	1.13	.890	.72	....	.08	.71	.65	.960	....
P_2O_5	6.64	6.317	6.50	6.46	6.33	6.30	6.49	6.560	6.450
SO_3	1.28	1.215	1.30	1.35	1.22	1.27	1.34	1.440	1.302
CO_2	15.65	15.355	15.86	....	....	15.65	15.15	....	15.533
Cl	.82	.82	.79	....	....	.82	.90	....	.83
Moisture	....	.310	....	.04	.16	....	.46	....	....
Carbon	.70	.319	....	....	[2]10.48	....	1.41	....	....
Total	100.63	99.845	97.44	....	....	95.86	....	....	....

[Other methods.]

	R. J. Davidson and T. L. Watson, Virginia.[3]	R. J. De Roode, West Virginia.[3]	R. J. Davidson and T. L. Watson, Virginia.[4]	Harry Snyder, Minnesota.[4]	W. P. Headden, Colorado.[4]	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Soluble SiO_2	1.01	4.31	1.15	....	5.05	....
Insoluble SiO_2	10.66	7.57	10.69	....	6.86	....
Total SiO_2	11.67	11.82	11.84	11.97	11.91	11.842
K_2O	....	25.78	26.01	25.72	25.18	25.673
Na_2O	....	.32	1.33	.86	1.66	....
CaO	....	27.26	27.57	26.79	26.49	27.028
MgO	....	7.36	7.50	7.41	7.15	7.355
Mn_3O_4	....	....	....	....	....	....
Fe_2O_3	....	.72	1.14	.86	.98	....
P_2O_5	....	6.60	6.68	6.25	6.43	6.49
SO_3	....	1.34	1.27	1.28	1.34	1.308
CO_2	....	20.28	14.77	15.75	....	....
Cl	....	....	.86	.84	....	.83
Moisture	....	....	....	....	....	....
Carbon	1.14	1.06	1.14	*.54*	1.36	1.175
Total	....	102.54	100.11	98.27	....	....

Figures in *italics* are not included in the averages.

[1] Dried at 110° C.

[2] Ignited to constant weight.

[3] Modification proposed by the reporter.

[4] Solution by treatment with HCl in beaker or dish and evaporation to dryness; analysis as usually performed in the laboratory of the chemist reporting.

Table II.

ASH SAMPLE NO. 4.

[Provisional method.]

	L. M. Bloomfield, Ohio.	R. J. de Roode, West Virginia.	F. L. Arnold, Massachusetts.	W. D. Bigelow, Department of Agriculture.	John Thompson, Minnesota.	Chas. Ryan, Colorado.	W. L. Rossman, Michigan.	Average.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Soluble SiO_2	18.950	3.500	....	....	....	15.59	....	....
Insoluble SiO_2	28.550	43.152	....	....	....	31.44	....	....
Total SiO_2	47.500	46.652	47.91	[1]*49.30*	46.64	47.03	47.53	47.21
K_2O	*12.370*	13.610	*14.18*	13.16	13.36	....	12.92	13.26
Na_2O	1.300	.446	....	.04	1.24	....	.46	....
CaO	12.690	12.770	*13.21*	....	12.79	*11.47*	....	12.75
MgO	7.940	8.436	....	....	8.12	*6.35*	....	8.165
Mn_3O_4	.440	....	....	....	....	.44	....	....
Fe_2O_3	1.780	1.547	....	.44	1.52	2.94	1.76	....
Al_2O_3	....	....	....	....	....	....	....	....
P_2O_5	8.620	8.500	8.25	8.29	8.48	8.39	8.44	8.424
SO_3	1.185	1.061	1.10	.99	1.00	1.09	1.16	1.083
CO_2	1.490	.700	....	....	2.70	2.84	....	....
Cl	.288	.190	....	....	.30	.75	....	....
Moisture	.625	.300	.54	.17	....	.69	....	....
Carbon	3.310	4.190	....	[2]5.06	....	3.76	....	....
Total	99.538	98.402	....	....	96.15	....	....	....

[Other methods.]

	R. J. Davidson and T. L. Watson, Virginia.[3]	R. J. de Roode, West Virginia.[4]	R. J. de Roode, West Virginia.[5]	Harry Snyder, Minnesota.[3]	W. P. Headden, Colorado.[3]	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Soluble SiO_2	.51	....	1.408	....	....	....
Insoluble SiO_2	46.55	28.13	45.500	....	....	....
Total SiO_2	47.06	....	46.908	46.95	47.22	47.035
K_2O	13.12	13.62	13.570	13.05	*12.54*	13.340
Na_2O	1.25	.51	.435	1.05	1.92	....
CaO	12.58	12.90	12.470	12.99	12.88	12.764
MgO	8.21	8.32	8.616	8.27	*5.65*	8.354
Mn_3O_4	....	....	....	....	.32	....
Fe_2O_3	1.97	1.547	1.547	1.66	3.31	....
Al_2O_3	1.08	....	....	....	....	....
P_2O_5	8.50	8.47	8.490	8.54	8.48	8.496
SO_3	1.07	1.03	1.030	1.12	1.14	1.078
CO_2	2.67	....	2.530	2.32	....	2.510
Cl	.30	....	....	.32	....	.310
Moisture	....	....	....	....	....	....
Carbon	3.04	2.22	....	4.62	3.40	....
Total	100.85	....	....	100.89	....	....

Figures in *italics* are not included in the averages. [1] Dried at 110°. [2] Ignited to constant weight.

[3] Treatment with HCl and evaporation to dryness; analysis as usual in the laboratory from which the report comes.

[4] Modification proposed by reporter. [5] From CO_2 portion analyzed by provisional method.

Table III.

SOIL SAMPLE No. 2.

[Provisional method; 36 hours' digestion.]

	L. M. Bloomfield, Ohio.	R. J. de Roode, West Virginia.	W. L. Rossman, Michigan.	[1] Harry Snyder, Minnesota.	[2] Harry Snyder, Minnesota.	[3] Harry Snyder, Minnesota.	[1] John Thompson, Minnesota.	[4] F. L. Arnold, Massachusetts.	F. T. Shutt, Canada.	L. Rossignol, Canada.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Insoluble matter	76.883	77.233	76.436	77.035	77.080	76.980	77.325	*78.18*	76.41	76.19
SiO_2 from HCl sol	....	.025	....	....	....	....	....	.22	.05	.04
K_2O	....	.397	.470	.275	.340	.450	.330	.49	.44	.48
Na_2O	....	.076	.155	.280	.140	.140	.125	....	....	....
CaO	....	.585	.521	.360	.410	.430	.430	.60	....	....
MgO	....	.519	.407	.360	.390	.400	.375	....	....	....
Mn_3O_4	....	.155	.275	....	....	....	....	....	....	....
Fe_2O_3	....	3.808	3.200	2.955	3.220	3.280	3.845	....	....	....
Al_2O_3	....	6.287	7.060	7.200	6.990	6.780	6.240	....	....	....
P_2O_5	.429	.480	.486	.580	.560	.530	.600	*.16*	.48	.47
Fe_2O_3, Al_2O_3, and P_2O_5	....	10.575	10.746	10.735	10.770	10.590	10.685	....	....	....
SO_3	....	.175	.175	.140	.120	.120	.105	....	....	....
CO_2	....	....	....	....	....	....	.100	....	....	....
Moisture at 100° to 110°	2.35	2.050	2.160	10.590	[10.590[	[10.590]	2.410	2.58	10.204	......
Organic and volatile	8.11	[4]8.383	8.325				8.130	7.30		......
Total	....	100.092	99.670	99.775	99.840	99.700	100.015	....	....	....
Nitrogen	.262	.270	.290	.270	....	....	....	....	.280	.282
P_2O_5, Goss's method	.425	.443	.487	....	....	....	....	....	....	....

Figures in *italics* are not included in the averages.

[1] Digestion in Snyder flask.

[2] In platinum flask and condenser.

[3] In platinum bottle, stoppered.

[4] Reported as dried at 110° C., figured to original soil by the reporter.

Table III—Continued.

SOIL SAMPLES No. 2—Continued.

[Provisional method; 36 hours' digestion.]

	Charles Ryan, Colorado.	E. E. Slosson, Wyoming.	W. D. Bigelow, Department of Agriculture.	D. Adriance, Texas.	E. Waller, New York City.	Arthur Goss, New Mexico.	A. M. Peter, Kentucky.	M. E. Jaffa, California.	W. M. Allen, North Carolina.	Average.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Insoluble matter	77.45	77.733	76.81	76.20	*78.23*	....	76.505	....	*74.82*	76.874
SiO_2 from HCl sol	....	.099	.092	.07	....	....	.022	....	....	.077
K_2O	.27	....	.40	.41	....	....	.510	....	....	.405
Na_2O	.02	....	.21	.32	....	....	.183	....	....	......
CaO	.52	.502	.38	.49	.577	....	.605	....	....	.460
MgO	.36	....	.40	.36	.589	....	.503	....	....	.425
Mn_3O_4	.16	.285	....	.45	....	....	.155	....	....	.247
Fe_2O_3	9.81	10.231	3.06	4.26	9.891	....	3.911	....	....	3.504
Al_2O_3			7.50	6.68		....	6.777	....	....	6.613
P_2O_5	.41	.494	.49	.46	.435	....	[1].537	....	.50	.496
Fe_2O_3, Al_2O_3, and P_2O_5	10.22	10.725	11.05	11.40	10.326	....	11.225	....	....	10.754
SO_3	.20	.141	.08	.15	....	....	.100	....	....	.137
CO_2	....	....	....	....	....	....	....	....	....	......
Moisture at 100° to 110°	2.69	....	2.31	*1.58*	....	....	2.423	....	....	2.372
Organic and volatile	*7.67*	....	[2]*9.44*	8.33	....	....	[2]8.217	....	....	8.249
Total	99.56	....	101.172	99.76	....	....	100.448	....	....	......
Nitrogen	....	....	.280[3]	....	....	....	....	....	....	.276
P_2O_5, Goss's method	....	....	....	....	....	.493	.486	*.68*	....	.467

Figures in *italics* are not included in the averages.

[1] Solution evaporated with HNO_3 before precipitating with molybdate at 40°C.

[2] Ignited to constant weight.

[3] Determined by T. C. Trescott.

Table IV.

SOIL SAMPLE No. 3.

[Provisional method; 36 hours' digestion.]

	L. M. Bloomfield, Ohio.	W. L. Rossman, Michigan.	[1] Harry Snyder, Minnesota	[1] John Thompson, Minnesota.	F. T. Shutt, Canada.	Le Rossignol, Canada.	Charles Ryan, Colorado.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Insoluble matter	81.225	80.186	81.015	81.045	79.98	80.12	80.21
SiO_2 from HCl sol					.06	.04	
K_2O		.451	.320	.305	.43	.44	.50
Na_2O		.205	.190	.130			.26
CaO		.375	.325	.310			.43
MgO		.381	.390	.310			*.79*
Mn_3O_4		.213					.18
Fe_2O_3		3.040	2.960	3.190			9.70
Al_2O_3		6.240	5.925	5.775			
P^2O_5	366	.376	.555	.505	.37	.37	.50
Fe_2O_3, Al_2O_3, and P_2O_5		9.656	9.440	9.470			10.20
SO_3		.193	.165	.075			.21
CO_2				.060			
Moisture at 100° to 110°	1.98	1.910	8.330	1.960	7.784		2.22
Organic and volatile	6.04	6.325		6.355			*5.76*
Total		100.895	99.175	100.020			100.86
Nitrogen	.175	.190	.180		.185	.188	
P_2O_5, Goss's method	.358	.375					

	W. D. Bigelow, Department of Agriculture.	D. Adriance, Texas.	E. Waller, New York City.	Arthur Goss, New Mexico.	A. M. Peter, Kentucky.	F. L. Arnold, Massachusetts.	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Insoluble matter	80.10	80.31	81.255		80.277	*82.48*	80.520
SiO_2, from HCl sol	.058	.05			.039	.21	.081
K_2O	.44	.42			.480	.43	.422
Na_2O	.20	.15			.167		
CaO	.30	.50	.388		.425	.36	.379
MgO	.27	.35	.524		.393		.381
Mn_3O_4		.39			.145		.232
Fe_2O_3	2.31	4.33	9.26		3.678		3.251
Al_2O_3	7.44	5.67			6.097		6.191
P_2O_5	.39	.35	*.28*		[2].445	.37	.418
Fe_2O_3, Al_2O_3, and P_2O_5	10.14	10.35	9.54		10.220		9.927
SO_3	.07	.11			.070		.128
CO_2							
Moisture at 100° to 110°	1.72	*.89*			1.855	*5.04*	1.941
Organic and volatile	[3]*7.12*	6.59			[3]6.395	*5.679*	6.341
Total	[4]100.418	100.11			100.466		
Nitrogen	.224						.190
P_2O_5, Goss's method				.390	.354		.369

Figures in *italics* are not included in the averages.

[1] Digestion in Snyder flask.

[2] Solution evaporated with HNO_3 before precipitating with molybdate at 40° C.

[3] Ignited to constant weight.

[4] Determined by Mr. T. C. Trescot.

Table V.

SOIL SAMPLE No. 2.

[Hilgard's method; 5 days' digestion in covered beakers.]

	L. M. Bloomfield, Ohio.	R. J. de Roode, West Virginia.	F. L. Arnold, Massachusetts.	W. L. Rossman, Michigan.	[1]Harry Snyder, Minnesota.	F. T. Shutt, Canada.	Le Rossignol, Canada.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Insoluble matter	77.910	75.380	*80.91*	76.350	77.015	76.18	75.84
Soluble SiO_2	....	9.330	....	....	....	....	....
K_2O	....	.543	.25	.477	.295	.42	....
Na_2O	....	.157	....	.594	.390	....	....
CaO	....	.630	....	.675	.400	....	....
MgO	....	.567	....	.627	.335	....	....
Mn_3O_4	....	.325	....	.300	....	....	....
Fe_2O_3	....	3.987	....	6.400	3.300	....	....
Al_2O_3	....	6.828	....	4.460	6.700	....	....
P_2O_5	.462	.473	.50	.480	.550	.51	....
Al_2O_3, Fe_2O_3, and P_2O_5	....	11.288	....	11.340	10.550	....	....
SO_3	....	.031	....	.309	.170	....	....
Moisture at 100° to 110°	10.46	2.050	....	2.035	10.590	....	....
Organic and volatile		8.383	6.96	7.140		....	....
Total	....	99.354	....	100.137	99.745	....	....
P_2O_5 in residue	.044	.037	....	....	....	....	....
Hygroscopic moisture	....	....	8.84	....	....	....	....

	M. E. Jaffa, California.	Chas. Ryan, Colorado.	E. E. Slosson, Wyoming.	W. D. Bigelow, Department of Agriculture.	D. Adriance, Texas.	A. M. Peter, Kentucky.	[2]Harry Snyder, Minnesota.	Average.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per cent.*	*Per cent.*	*Per cent.*
Insoluble matter	*73.99*	77.17	77.775	75.62	76.28	76.880	77.03	76.619
Soluble SiO_2	8.60	8.31	9.795	....	....	....	....	9.008
K_2O	.67	.41	....	.65	.36	.382	.28	.431
Na_2O	.15	.32	....	.13	.32	.215	.19	....
CaO	.43	.68	.533	.54	.54	.565	.39	.538
MgO	.39	.32	....	.45	.34	.478	.32	.425
Mn_3O_4	*.05*	*.06*	....	....	.32	.135	....	.295
Fe_2O_3	5.19	10.13	10.248	10.89	4.30	3.950	3.30	3.347
Al_2O_3	6.25				6.53	6.527	6.70	6.285
P_2O_5	.66	.43	.511	.51	.47	[3]*.378*	.56	.510
Al_2O_3, Fe_2O_3, and P_2O_5	12.10	10.56	10.759	11.40	11.30	10.855	10.56	11.071
SO_3	.04	.15	....	.14	.17	.095	.13	.135
Moisture at 100° to 110°	[4]5.24	2.51	10.091	2.31	9.77	2.423	....	....
Organic and volatile	6.85	7.76		[5]9.44		[5]8.217	....	....
Total	99.91	99.93	....	100.66	99.40	100.245	....	....
P_2O_5 in residue	....	....	....	None..	....	.067	....	.047
Hygroscopic moisture	8.62	4.23	....	....	....	....	....	....

Figures in *italics* are not included in the averages.

[1] Solution analyzed by the Provisional method.

[2] Digestion in platinum beaker; solution analyzed by Provisional method.

[3] Residue still red from Fe_2O_3 after 2 days' digestion.

[4] Drying 30 minutes at 200° C.

[5] Ignited to constant weight.

Table VI.

SOIL SAMPLE NO. 3.

[Hilgard's method; 5 days' digestion in covered beakers.]

	L. M. Bloomfield, Ohio.	[1] Harry Snyder, Minnesota.	[1] John Thompson, Minnesota.	F. T. Shutt, Canada.	Le Rossignol, Canada.	M. E. Jaffa, California.	Chas. Ryan, Colorado.	W. D. Bigelow, Department of Agriculture.	D. Adriance, Texas.	A. M. Peter, Kentucky.	F. L. Arnold, Massachusetts.	Average.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Insoluble matter	82.01	81.445	80.89_5	80.10	79.74	79.47	80.17	79.62	80.31	80.720	*83.85*	80.448
Soluble silica	2.54	......	......	......	......	8.08	6.91	......	......	......	......	......
K_2O	......	.310	.310	.43	......	.46	.33	.63	.47	.388	.24	.396
Na_2O	......	.295	.130	......	......	.10	.08	.13	.10	.223	......	......
CaO	......	.310	.275	......	......	.39	.60	.37	.60	.420	.32	.411
MgO	......	.265	.330	......	......	.30	.49	.33	.44	.431	......	.369
Mn_3O_4	......	......	......	......	......	.04	.08	......	.32	.115	......	.139
Fe_2O_3	......	3.025	3.200	......	......	4.87	9.86	10.21	4.25	3.386	......	3.746
Al_2O_3	......	5.975	5.890	......	......	5.05			5.77	6.164	......	5.770
P_2O_5	.309	.550	.560	.44	......	.40	.48	.38	.33	[4] *.285*	.41	.429
Fe_2O_3, Al_2O_3 and P_2O_5	......	9.550	9.650	......	......	10.32	10.34	10.59	10.35	9.835	......	10.091
SO_3	......	.140	.130	......	......	.06	.13	.11	.18	.065	......	.116
Moisture at 100° to 110°	8.02	8.242	1.960	......	......	[3] 3.37	2.28	1.72	7.45	1.855	......	......
Organic and volatile			6.355	......	......	5.51	5.70	[2] 7.12		[2] 6.395	5.12	......
Total	......	100.560	100.890	......	....	100.02	100.20	100.62	100.22	100.447	......	......
P_2O_5 in residue	.032	......	......	......	......	......	......	None.	......	.067	......	......
Hygroscopic moisture	......	......	......	......	......	6.51	5.61	......	......	......	......	......

Figures in *italics* are not included in the averages.

[1] Solution analyzed by the Provisional method.
[2] Ignited to constant weight.
[3] At 200°.
[4] Insoluble residue still red with Fe_2O_3 after 2 days' digestion.

Table VII.

[Digestion for different times. Solutions analyzed by the provisional method.]

SOIL SAMPLE No. 2.

	Digestion for 2 hours.						
	R. J. de Roode, West Virginia.[1]	W. L. Rossman, Michigan.[1]	D. Adriance, Texas.[1]	F. T. Shutt, Canada.[1]	F. T. Shutt, Canada.[2]	W. M. Allen, North Carolina.[1]	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Insoluble matter	79.896	79.380	79.30	79.400	80.610	80.61	79.866
K_2O	.208	.251	....	.245	.144	....	.212
Na_2O	.050	.077	....	....	....	....	....
CaO	.580	.450	....	....	....	....	....
MgO	.391	.279	....	....	....	....	....
Mn_3O_4	.275	.240	....	....	....	....	....
Fe_2O_3	3.570	2.560	....	....	....	....	....
Al_2O_3	4.480	5.926	....	....	....	....	....
P_2O_5	.425	.454	....	.453	.435	.41	.435
Fe_2O_3, Al_2O_3, and P_2O_5	8.475	8.940	....	....	....	....	8.708
SO_3	.074	.137	....	....	....	....	....

	10 hours.		24 hours.	36 hours.[3]			5 days.
	A. M. Peter, Kentucky.[1]	A. M. Peter, Kentucky.[4]	W. M. Allen, North Carolina.[1]	A. M. Peter, Kentucky.[1]	W. M. Allen, North Carolina.[1]	Average from Table III.	Average from Table V.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Insoluble matter	77.434	77.362	76.68	76.527	74.82	76.874	76.619
K_2O	.356	.358	....	.510	....	.405	.431
Na_2O	....	....	....	.183	....	....	....
CaO	....	....	....	.605	....	....	....
MgO	....	....	....	.503	....	....	....
Mn_3O_4	....	....	....	.155	....	....	....
Fe_2O_3	4.097	4.087	....	3.911	....	3.504	4.347
Al_2O_3	5.672	5.678	....	6.777	....	6.613	6.285
P_2O_5	[5].521	[5].480	....	[5].537	.50	.496	.510
Fe_2O_3, Al_2O_3, and P_2O_5	10.290	10.245	....	11.225	....	10.754	11.071
SO_3	....	....	....	.100	....	....	....

[1] Digestion in stoppered bottle.

[2] In covered beaker.

[3] Soil 2, 36 hours' digestion in flask covered and set on bath, insoluble matter equals 76.915; in flask covered and immersed to neck, insoluble matter equals 76.614; in flask with condensing tube and immersed to neck, insoluble matter equals 76.406.

[4] In flask with condensing tube.

[5] Solution evaporated with HNO_3 before precipitating with molybdate at 40° C.

Table VII—Continued.

[Digestion for different times. Solutions analyzed by the Provisional method.]

SOIL SAMPLE No. 3.

	Digestion for 2 hours.						
	L. M. Bloomfield, Ohio.[1]	W. L. Rossman, Michigan.[1]	D. Adriance, Texas.[1]	F. T. Shutt, Canada.[1]	F. T. Shutt, Canada.[2]	W. M. Allen, North Carolina.[1]	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Insoluble matter	85.01	83.090	83.75	82.770	83.956	84.03	83.519
K_2O	....	.201	....	.164	.152	....	.172
Na_2O	....	.120	....	....	....	....	....
CaO	....	.301	....	....	....	....	....
MgO	....	.215	....	....	....	....	....
Mn_3O_4	....	.150	....	....	....	....	....
Fe_2O_3	....	1.920	....	....	....	....	....
Al_2O_3	....	5.359	....	....	....	....	....
P_2O_5	....	.351	....	.422	.415	.325	.378
Fe_2O_3, Al_2O_3, and P_2O_5	....	7.630	....	....	....	....	....
SO_3	....	.198	....	....	....	....	....

	10 hours.		24 hours.	36 hours.			5 days.
	A. M. Peter, Kentucky.[1]	A. M. Peter, Kentucky.[3]	W. M. Allen, North Carolina.[1]	A. M. Peter, Kentucky.[1]	W. M. Allen, North Carolina.[1]	Average from Table IV.	Average from Table VI.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Insoluble matter	81.031	81.165	81.21	80.316	80.39	80.520	80.448
K_2O	.324	.332	....	.480	....	.422	.396
Na_2O	....	....	....	.167	....	....	....
CaO	....	....	....	.425	....	....	....
MgO	....	....	....	.393	....	....	....
Mn_3O_4	....	....	....	.145	....	....	....
Fe_2O_3	3.406	3.464	....	3.678	....	3.251	3.746
Al_2O_3	5.583	5.401	....	6.097	....	6.191	5.770
P_2O_5	[4].406	[4].365	....	[4].445	....	.418	.429
Fe_2O_3, Al_2O_3, and P_2O_5	9.395	9.230	....	10.220	....	9.927	10.091
SO_3	....	....	....	.070	....	....	....

[1] Digestion in stoppered bottle.
[2] In covered beaker.
[3] In flask with condensing tube.
[4] Solution evaporated with HNO_3 before precipitating with molybdate at 40° C.

Table VII—Continued.

(Digestion for different times. Solutions analyzed by the Provisional method.)

SOIL SAMPLE No. 5.

	10 hours.		36 hours.
	A. M. Peter, Kentucky.[1]	A. M. Peter, Kentucky.[2]	A. M. Peter, Kentucky.[1]
	Per cent.	*Per cent.*	*Per cent.*
Insoluble matter	85.090	84.860	83.901
K_2O	.392	.410	.601
Na_2O	....	....	.159
CaO	....	....	.220
MgO	....	....	.440
Mn_3O_4	....	....	.210
Fe_2O_3	3.357	3.483	3.464
Al_2O_3	5.173	5.090	5.638
P_2O_5	[4].105	[4].102	[4].138
Fe_2O_3, Al_2O_3, and P_2O_5	8.635	8.675	9.240
SO_3	....	....	.032

[1] Digestion in stoppered bottle.

[2] In flask with condensing tubes.

[4] Solution evaporated with HNO_3 before precipitating with molybdate at 40° C.

SAMPLE No. 115.

	2 hours.	24 hours.	36 hours.
	W. M. Allen, North Carolina.	W. M. Allen, North Carolina.	W. M. Allen, North Carolina.
	Per cent.	*Per cent.*	*Per cent.*
Insoluble matter	93.02	91.58	91.96
K_2O	....	....	....
Na_2O	....	....	....
CaO	....	....	....
MgO	....	....	....
Mn_3O_4	....	....	....
Fe_2O_3	....	....	....
Al_2O_3	....	....	....
P_2O_5	....	....	....
Fe_2O_3, Al_2O_3, and P_2O_5	....	....	....
SO_3	....	....	....

Table VIII.

TABLE OF AVERAGES.

SOIL SAMPLE No. 2.

	Provisional method.					Hilgard method.				
	No. included.	Average.	Highest.	Lowest.	Difference in per cent of average.	No. included.	Average.	Highest.	Lowest.	Difference in per cent of average.
		Per ct.	*Per ct.*	*Per ct.*			*Per ct.*	*Per ct.*	*Per ct.*	
Insoluble matter	14	76.874	77.733	76.19	2	12	76.619	77.910	75.380	3.3
K_2O	13	.405	.510	.27	59	11	.431	.670	.250	98
CaO	13	.460	.605	.360	53	10	.538	.680	.390	54
MgO	11	.425	.589	.360	54	9	.425	.627	.320	72
Fe_2O_3	9	3.504	4.26	2.955	37	7	4.347	6.400	3.300	70
Al_2O_3	9	6.613	7.500	6.240	19	7	6.285	6.828	4.460	38
P_2O_5	16	.496	.600	.410	38	12	.510	.660	.430	45
Fe_2O_3, Al_2O_3, and P_2O_5	12	10.754	11.400	10.220	11	10	11.071	12.100	10.550	14
Nitrogen	7	.276	.290	.262	10					
P_2O_5, Goss method	5	.467	.493	.425	15					

SOIL SAMPLE No. 3.

	Provisional method.					Hilgard method.				
	No. included.	Average.	Highest.	Lowest.	Difference in per cent of average.	No. included.	Average.	Highest.	Lowest.	Difference in per cent of average.
		Per ct.	*Per ct.*	*Per ct.*			*Per ct.*	*Per ct.*	*Per ct.*	
Insoluble matter	11	80.520	81.255	79.980	1.6	10	80.448	82.010	79.47	3.1
K_2O	10	.422	.500	.305	46	9	.396	.630	.240	98
CaO	9	.379	.425	.300	33	8	.411	.600	.275	79
MgO	8	.381	.524	.270	67	7	.369	.490	.265	61
Fe_2O_3	6	3.251	4.330	2.310	62	5	3.746	4.870	3.025	49
Al_2O_3	6	6.191	7.440	5.670	29	5	5.770	6.164	5.050	19
P_2O_5	11	.418	.555	.350	49	9	.429	.560	.309	59
Fe_2O_2, Al_2O_3, and P_2O_5	8	9.927	10.350	9.440	9	7	10.091	10.590	9.550	10
Nitrogen	6	.190	.224	.175	26					
P_2O_5, Goss method	4	.369	.390	.354	10					

Table IX.

ACTION OF HCl ON GLASS AND PORCELAIN VESSELS.

	Arnold, Mass.	Snyder, Minn.	Shutt, Canada.	Slosson, Wyoming.	Bigelow, Dept. Agr.	Ryan, Col.	Peter, Ky.	R. J. de Roode, W. Va.	Average.
	Grams.	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grms.*
Residue from glass vessel	.0028	.0012	.0023	.0014	.0017	.0016	.0008	.0019	.0017
Residue from porcelain vessel	.0019	.0037	None.	.0011	.0028	.0022	.0011	.0020	.0021
Kaehler & Martini's Apparatenglas					.0012				

REMARKS AND EXTRACTS FROM REPORTS.

Mr. DAVIDSON, Virginia. The method which we use here is to weigh out 2 grams ash into a porcelain dish, treat with HCl, and evaporate to dryness. This operation is repeated. Then we add HCl and hot water, allow to stand about half an hour, warm and filter through a weighed filter, wash until free from chlorides. The filtrate and washings are run into a 200 cc. flask. The residue is put into a steam bath and heated to constant weight, then burned to whiteness and the carbon estimated. The residue is then transferred to a platinum dish and boiled with Na_2CO_3 solution, filtered and washed and the insoluble SiO_2 determined, the soluble calculated by difference. * * * The filtrate is made up to 200 cc. and three portions of 50 cc. taken out—one for Fe_2O_3, CaO and MgO; one for P_2O_5; one for SO_3 and alkalies. The first portion is heated, some sodium phosphate added, and then oxidized with $KClO_3$, and heating continued until Cl is driven off, cooled, made alkaline with ammonia, then acid with acetic acid, warmed and filtered, the residue dissolved in HCl and reprecipitated, filtered, washed, dried, burned, and weighed as phosphates of Fe and Al. This is dissolved and Fe determined by permanganate solution. The Al is determined by difference. The CaO and MgO are determined by usual methods in the filtrate from the phosphates of Fe and Al, precipitating the Ca first as oxalate from the hot solution.

In the second portion of 50 cc. the P_2O_5 is determined by the molybdate method and in the third the SO_3 and alkalies by $BaCl_2$ and the $Ba(OH)_2$ method. Cl was determined volumetrically in a water solution of the ash and CO_2 was weighed by absorption in potash bulbs. In the analysis by the Provisional method the same portion was used for carbon, CO_2 and Cl. Dr. Davidson does not like the method for C and SiO_2 proposed by the reporter, as the filtration was slow and difficult.

L. M. BLOOMFIELD, Ohio. Found carbon in the ash unsatisfactory. A number of duplicates on ash No. 4 differed widely. The figure given is a mean of the three that agreed most closely. Soluble SiO_2 was separated by boiling one-half hour with very strong solution of Na_2CO_3, replacing water as it evaporated and adding a few drops of NaOH after the boiling. Mr. Bloomfield likes the Goss method for P_2O_5.

Mr. R. J. DE ROODE, West Virginia. In regard to drying soil in a tube at 200° C. Dr. de Roode says that he "could get no constant weight after successive half-hour dryings at 200° C. A constant decrease in weight took place. * * * All efforts to get constant moisture determinations were failures except that of drying in a water-oven. This gives good results, and I think we should adopt it. The Hilgard method of drying in a tube at 200° C. was the worst of all." Ash No. 1 gained weight on drying at 110° and also on ignition. The method for C and SiO proposed by the reporter was not found satisfactory. "The filtration on a Gooch with a perforated disk is very slow, and the removal of the residue and boiling with Na_2CO_3 quite unsatisfactory. The solution of Na_2CO_3 upon acidifying and evaporating for SiO_2 gave up a small amount of carbon, which was mixed with the silica and hence was burned and lost." Dr. de Roode does not like the Goss method for P_2O_5 in soils, and thinks small quantities of P_2O_5 are not completely precipitated.

F. L. ARNOLD, Massachusetts. The figures are the average of two closely agreeing tests in all cases, instructions being carefully followed.

HARRY SNYDER and J. THOMPSON, Minnesota. The ash analyses by Mr. Thompson were by the Provisional methods; those by Mr. Snyder were by the usual methods followed in the laboratory: "By first determining CO_2 by KOH bulbs, then transferring to a covered beaker, boiled two hours, evaporated to dryness, and then the analysis made in the usual way. The carbon was determined in another portion by dissolving in dilute acid, filtering on a weighed filter, drying, weighing and then igniting. In all other operations the provisional methods were followed." In the analyses of the soils, the insoluble matter includes the SiO_2 separated from the HCl solution. CaO was determined volumetrically by permanganate solution. P_2O_5 was determined in a fresh portion of the original acid solution. Difficulty was experienced in perfectly separating the insoluble matter on account of fine particles passing through the filter. "The analyses reported were all made in duplicate at different times during the year." The duplicate analyses reported agree very closely in every instance. Mr. Snyder also made a series of digestions in platinum vessels, as noted in the tables of analyses. His conclusion, from the results obtained, is that "with these samples of soils, which are characteristic of this class, there does not appear to be any material difference in the results whether a porcelain beaker or a glass digestion flask is used, the results in both cases being practically the same as those obtained by using platinum ware for the digestion."

Mr. FRANK T. SHUTT and Mr. LE ROSSIGNOL, Canada. Nitrogen was determined (*a*) by the Gunning-Kjeldahl method, modified for nitrates, and (*b*) by the Scovell-Kjeldahl modification. The figures reported are the means of two or more determinations which differed only in the third decimal. In the soil digestions the water bath was heated day and night. In testing the action of HCl on glass and porcelain the acid had to be renewed three times in the glass and eight times in the porcelain vessel.

Mr. M. E. JAFFA, California. The P_2O_5 in No. 2 is a mean of two independent determinations, giving respectively 0.66 and 0.68 per cent. Mr. Jaffa reports also "humus ash" in soil No. 2, 2.42 per cent, containing 0.19 P_2O_5; in soil No. 3, 2.17 per cent, containing 0.17 P_2O_5.

Messrs. WM. P. HEADDEN and CHAS. RYAN, Colorado. Much trouble was experienced with the ash samples, especially SiO_2 in No. 4, and the results were not considered satisfactory. Moisture in the ashes was determined by drying in the water oven.

Mr. W. D. BIGELOW, U. S. Department of Agriculture. The time allowed for the formation of the molybdate precipitate (in the Goss method) is not sufficient, and the temperature at which it is precipitated is too high. Have not succeeded in getting results which would enable one to form any opinion concerning the method of extraction.

Mr. ELWYN WALLER, New York City, reports insoluble matter from treatment by Goss's method for P_2O_5 in soil No. 2, 75.582, and No. 3, 79.367. Potash from this solution 0.263 and 0.183, respectively. Mr. Waller expresses himself as not satisfied with his results, having been much hurried with the work. He also notes the presence of Mn in his Ca and Mg precipitates, it not having been previously separated.

Mr. ARTHUR GOSS, New Mexico, remarks that, in using his method, it is very important that the molybdic solution be of full strength. Old solutions that have deposited some of their molybdic acid will not give good results. He also reports CaO 0.37 and K_2O 0.51 in sample No. 3 by his method, and P_2O_5 0.086 in the reporter's soil sample No. 5.

Mr. A. M. PETER, Kentucky. The Provisional method was not followed in the case of P_2O_5. Instead, the aliquot representing 2 grams of soil was evaporated to dryness, heated with conc. HNO_3, diluted, filtered from a few flakes of silica, and precipitated with molybdic solution and let stand over night in a warm place not exceeding 40° C. The NH_4HO precipitates were always redissolved and reprecipitated, as was also the precipitate by oxalate of ammonia in the CaO determination. In the Hilgard method for P_2O_5 it was impossible to dissolve the ignited soil by two days' digestion in HNO_3, the residue still being red. This accounts for the low results reported. The CaO obtained by the Hilgard method was brown from the presence of Mn_3O_4. This was separated by solution of the CaO in acetic acid.

THE ASH ANALYSES.

The results in Tables I and II show a reasonably good agreement, though not all that could be desired, except in the case of carbon, silica and sand, and carbon dioxide, where the variations are such as to indicate that the methods followed are utterly unreliable. The determinations of carbon and sand are important in the analysis of the ashes of plants for the reason that it is desirable to determine the composition of the pure ash by excluding from the analysis carbon, sand, and carbon dioxid, and for this reason your reporter selected these determinations as the principal ones to be investigated this year. It was evident to him at the outset that these determinations could not be made with any degree of certainty by the methods adopted provisionally by this association, these methods evidently not having been intended in the first place to cover the analysis of the ashes of plants in the strict sense of that expression. These opinions are fully confirmed by the analyses reported. There is evidently an utter failure to accomplish the solution of the "soluble SiO_2" in the Na_2CO_3 solution, especially in the case of ash No. 4, in which the proportion of silica is very large. Your reporter has tried to throw some additional light upon the question of the separation of the "soluble SiO_2" by working upon an ash which was known to be practically free from silica in any form except that properly belonging to the plant itself. The following investigation was carried out with the assistance of Mr. B. C. Keiser:

To obtain an ash which should be as entirely free from actual sand and soil as possible, the internodes of cornstalks were used as having a polished surface which could be freed perfectly from adhering dirt. These were washed, dried, cut in slices, and burned at the lowest possible temperature in a platinum dish and the ash sifted and bottled. Six portions of this ash, of 0.5 gram each, were weighed out and treated with 10 cc. HCl each and evaporated to dryness to render SiO_2 insoluble, redissolved, filtered, and washed in the usual way, and the residues, consisting of carbon, silica, and sand, were treated as follows:

Two residues were washed from the filters while still moist into a platinum dish, enough Na_2CO_3 added to make about a 10 per cent solution, boiled two or three minutes, decanted through the same filters, and the residues boiled again with fresh Na_2CO_3 solution a second and a third time. The residue was then brought upon the filter and washed with hot water, then with a little dilute HCl, and finally with hot water again. The residues, after ignition, weighed 0.0022 and 0.0020 gram, equivalent to 0.44 and 0.40 per cent "sand" in the ash.

Two residues, treated in a similar manner, using 10 per cent NaOH solution instead of Na_2CO_3 solution, gave 0.0024 and 0.0018 gram insoluble, equivalent to 0.48 and 0.36 per cent "sand" in the ash.

Two residues were ignited without other treatment and weighed 0.0659 and 0.0655 gram, equivalent to 13.18 and 13.10 per cent of "sand and silica."

The first of these residues was then treated as before described, and gave 0.0345 gram or 6.90 per cent insoluble in the Na_2CO_3 solution, and upon repeating the treatment 0.0284 gram or 5.68 per cent still remained.

The second residue was treated in like manner with 10 per cent NaOH solution, giving 0.0231 gram or 4.62 per cent insoluble, and on repeating the treatment 0.0181 gram or 3.62 per cent still remained.

These results show that the silica which was readily soluble before ignition in solutions of sodium carbonate or hydrate are only slowly dissolved when the silica has been ignited. In this case boiling six times with the alkaline solvent failed to effect the separation. The same thing is evident in the results on ash sample No. 4 in Table II. It would be absurd to suppose that the ash really contained more than 5 per cent of actual sand or soil. The calculation of the "pure ash" based on any of the findings of "sand" reported in Table II would have been entirely erroneous.

The action of caustic alkali is a little stronger than that of the carbonate, but it would probably be best to continue the use of the carbonate and insist that the separation be made before the ignition of the residue. The same remark will, of course, apply to this determination in the residue from a soil.

The conclusion to be drawn is, clearly, that the separation of the silica ought to be effected before the residue is ignited. As hydrated silica still retains moisture after drying at 100 or 110°, it is also evident that the carbon determination, depending upon the loss of weight of the siliceous residue after drying at 110°, must be subject to a considerable error when the residue is very siliceous. The method suggested by your reporter for trial was intended to obviate these difficulties, but was not entirely satisfactory. However, the principles above stated are certainly correct, and your reporter recommends that they be observed in any scheme of analysis that is adopted and respectfully presents the appended sketch of a method of ash analysis for your consideration.

THE SOIL ANALYSES.

The results of the soil analyses show wide and discouraging differences between the findings by different chemists, though, in the majority of instances, the duplicates reported by the same chemist agree closely. The only determinations in which a fair agreement has been obtained are those of the total insoluble matter, the ammonia precipate, and the nitrogen. The agreement of results on P_2O_5 by the method proposed by Mr. Arthur Goss is better than that of results by other methods, but the number of results reported is comparatively few. The discrepancies occur even in those determinations in which a good concordance was naturally to be expected, as, for instance, those of iron and lime. These wide variations, considered in connection with the comparatively good agreement in the total insoluble matter, seem to point to a lack of uniformity in carrying out the method of analysis after the soil solution has been obtained. Your reporter has not concerned himself so much with the method of analysis of the soil solution as with that of the digestion, or how the solution is to be obtained, this being the prominent question to be decided this year. Involved with this question is that of the material of the vessel in which the digestion is to be performed, and it is hoped that the results reported in Table VIII, together with those of Snyder, of Minnesota, who made a series of analyses in platinum vessels, may put an end to the doubt that seems to exist in the minds of some by showing that good glass is fully equal to porcelain, and probably somewhat superior to it, for this purpose. The comparison of poor glass and porcelain need not be drawn, as in these days there is no excuse for using anything but the best in this kind of work. Whether glass or porcelain be used, the amount extracted from the vessel is so small that it is utterly insignificant.

METHOD OF DIGESTION.

Your reporter had to consider practically three methods of soil digestion: the beaker method, the digestion in closed vessels, and the digestion in vessels which were open to the atmospheric pressure, but in which evaporation is reduced to a minimum by a condensing arrangement, as proposed by Snyder. The time of digestion also entered into the question to be considered. The end to be obtained is uniformity and accuracy of results. The hasty inspection which your reporter has been able to make of the results herewith tabulated seems to show a distinctly greater degree of uniformity in the results by the provisional method of thirty-six hours' digestion in closed vessels than by the Hilgard method; also more uniformity in the two-hour digestions than where the digestion was for thirty-six hours, though in this case we have not so many results for comparison, and the difference may be accidental.

An inspection of the table of averages will show that the range of variation, while much too great in both methods, is distinctly greater in the Hilgard than in the Provisional methed, notwithstanding the fact that we are considering more reports by the latter method than by the former. Lack of familiarity with the Hilgard method can hardly be the cause of this, as some of the largest variations are found in the reports of those who should be most familiar with the method.

These differences appear even in the determinations of insoluble matter and the iron and alumina precipitate, which are the determinations best calculated to show differences dependent on the digestion. It is also evident that, as the digestion is prolonged, the decomposition of silicates, principally of alumina and potash, continues, as was pointed out by the reporter for 1892. In some cases the agreement of results of different methods carried out in the same laboratory has been very close. A remarkable instance of this is found in the work of Snyder and Thompson, of Minnesota, in which the agreement in the insoluble matter obtained by closed digestion, beaker, and Snyder flash is so close as to suggest that some subsequent treatment, possibly a slow evaporation of the acid solution upon the residue, may have practically reduced the three series to one method.

The main points in the argument of the reporter of last year in support of the beaker method of digestion seem to be: (1) in covered beakers the acid is soon brought by evaporation to constant strength, because it is well known that pure hydrochloric acid, when evaporated under atmospheric pressure, tends to reach a constant strength; (2) the action of HCl of sp. gr. 1.115 on soils is practically exhausted after five days and is practically the same for five as for ten days' digestion; (3) it is not desirable to throw out of comparison with our work the great number of analysis that have been made by the beaker method, and fourth, the results of the beaker method have been successfully interpreted in accordance with agricultural practice and experience.

In regard to the first point, it is evident at once that the conditions in the digestion of a soil are not the same as those attending the evaporation of pure acid, unless the soluble matter of the soil is so small in comparison with the amount of acid used that it may be regarded as nothing. This is rarely the case, unless in the sands of Florida and similar regions. As soon as any matter is dissolved from the soil the solution becomes less capable of retaining the gaseous HCl. The conditions, therefore, instead of being uniform will vary with the amount of soluble matter in the soil and the amount of evaporation, and as the amount of evaporation during five days' digestion from a beaker covered with a watch glass is by no means a constant quantity, we have evidently lost control over one of the conditions which it was in our power to regulate. To throw a little more light on this point, the following experiments were made, with the assistance of Mr. B. C. Keiser, of the Kentucky State College laboratory, to determine the amount of free HCl in a given volume of the acid liquid during the digestion of a soil by different methods.

This determination was made by drawing out with a small, accurate pipette two portions of 1 cc. each from the solution to be tested. One of these portions was neutralized with normal NaOH solution, the result being put down as "total acidity;" the other portion was evaporated to dryness on the water bath, taken up with a little water and evaporated again to dryness, then taken up with water and neutralized like the first portion, the result being put down as "acidity of salts," or, for the sake of brevity, "combined acid;" the difference gives the "free acid." In one experiment by the five-day digestion on the water bath, the solution was tested each morning, except on the fifth day, where one had evaporated too low to admit of the portions being withdrawn. In the other experiments the test was made only at the end of the digestion. The figures in the table represent the number of cubic centimeters of normal NaOH required to neutralize 1 cc. of solution. One cubic centimeter of the HCl, sp. gr. 1.115, used required 7.05 cc. NaOH. An experiment with $CaCO_3$ instead of soil was made also, and this is included in the table for further illustration. The results show, as was to be expected, a slight gain in strength at first, never reaching that of the original acid, followed by a gradual decrease in acidity as the evaporation is carried further.

Table X.

STRENGTH OF ACID DURING SOIL DIGESTION.

Time of digestion.		First day.	Second day.	Third day.	Fourth day.	Fifth day.	36 hours.
Two grams of soil + 20 cc. HCl, 1.115 sp. gr., in porcelain beaker, covered with a watch glass, heated about twelve hours each day on the water bath:		*cc. alk.*	*cc. alk.*	*cc. alk.*	*cc. alk.*	*cc. alk.*	*cc. alk.*
Soil No. 2	Total acid	6.80	6.90	6.95	6.75	(*)	......
	Combined acid	.35	.45	.60	......		......
	Free acid	6.45	6.45	6.35	......		......
Soil No. 3	Total acid	6.90	6.95	6.95	6.75	6.95	......
	Combined acid	.35	.35	.50	.50	.60	......
	Free acid	6.55	6.60	6.45	6.25	6.35	......
Soil No. 5	Total acid	6.95	6.90	7.00	6.90	7.00	......
	Combined acid	.35	.35	.45	.50	.60	......
	Free acid	6.60	6.55	6.55	6.40	6.40	......
Soil No. 2	Total acid	......	......	......	......	7.00	......
	Combined acid	......	......	......	......	.50	......
	Free acid	......	......	......	......	6.50	......
Soil No. 3	Total acid	......	......	......	......	7.00	......
	Combined acid	......	......	......	......	.45	......
	Free acid	......	......	......	......	6.55	......
Soil No. 5	Total acid	......	......	......	......	6.90	......
	Combined acid	......	......	......	......	.45	......
	Free acid	......	......	......	......	6.45	......
5 grams soil 2 + 50 cc. HCl in an Erlenmeyer flask simply covered and set on the bath:	Total acid	......	......	......	......	......	6.93
	Combined acid	......	......	......	......	......	.50
	Free acid	......	......	......	......	......	6.43
5 grams soil 2 + 50 cc. HCl in pressure bottle in the water bath.	Total acid	......	......	......	......	......	6.73
	Combined acid	......	......	......	......	......	.50
	Free acid	......	......	......	......	......	6.23
5 grams soil 2 + 50 cc. HCl in Erlenmeyer flask with condensing tube, immersed in the bath.	Total acid	......	......	......	......	......	6.68
	Combined acid	......	......	......	......	......	.45
	Free acid	......	......	......	......	......	6.23
5 grams soil 2 + 50 cc. HCl in Erlenmeyer flask simply covered and immersed to the neck in bath.	Total acid	......	......	......	......	......	6.83
	Combined acid	......	......	......	......	......	.50
	Free acid	......	......	......	......	......	6.33
1 gram $CaCO_3$ + 25 cc. HCl in covered porcelain beaker on water bath: Free acid		6.35	6.40	6.45	6.45	6.43	......
Same as the preceding but uncovered until about 15 cc. had evaporated: Free acid		5.60	......	......	......	......	......
25 cc. HCl without addition in covered porcelain beaker: Free acid		7.15	7.13	7.13	7.13	7.15	......

* All evaporated.

In regard to the second point, while our results do not cover a period of digestion longer than five days, it appears that for the times of digestion examined the action of the acid upon the silicates continues, and there is no reason that there is a limit upon this action within the period of five days. The figures referred to by the reporter of last year in support of his position (Am. Jour. Science, Jan. 1874, p. 20) can hardly be considered as supporting it unless we are willing to admit that the experimental errors of the method employed are very considerable. For example, the total insoluble matter found after one day's digestion was 85.57 per cent, and after three days, 83.84 per cent, showing that 1.73 per cent of the original soil had been dissolved as a result of the second and third day's digestion. On the fourth day the total insoluble matter was 83.50, and on the fifth day, 82.83 per cent, showing that 0.67 per cent of the original soil had been dissolved as the result of the fifth day's action, or more than a third as much as was dissolved in the former period of two days. The residue reported as obtained by ten day's digestion, however, is 82.73 per cent, practically the same as obtained by five days' digestion. A similar comparison of the amounts of "soluble bases" reported shows that during the fifth day of the digestion about one-fourth as much was dissolved as in the period of two days between the first and third days, and that by ten days' digestion the same amount of bases was dissolved as by three days' digestion. The plain construction to be placed upon these figures is that the experimental errors of the method were sufficient to obscure the solvent action that was actually taking place.

In regard to the third point, it does not seem impossible to establish a basis of comparison between any method yielding fairly uniform results and the results of the beaker method, and this should certainly not bar us from the adoption of a method that would yield concordant results in the hands of different chemists. Your reporter does not desire to depreciate in the least the value of the work that has already been done. On the contrary, he desires to place himself on record as holding in the highest appreciation the work of the pioneers of soil analysis in this country. But theirs was the work of pioneers and the difficulties in their way were similar to those that pioneers in every field have met and overcome according to the limited means at their disposal. It is our duty to cultivate with the best of modern appliances the fields which they have opened, and we would be very remiss if we did not constantly amend our methods until they were brought to the greatest degree of refinement consistent with our present knowledge. Lastly, in regard to the matter of interpretation of results, it is self-evident that the more concordant the results are among themselves the more readily will they be interpreted and the more surely will the conclusions reached be in accordance with actual practice. If the system of interpretation is a true one, the elimination of experimental errors will certainly involve no change in that system.

In view of all the considerations brought out, your reporter does not consider it advisable to adopt the beaker method of digestion, nor the method, as a whole, proposed by the reporter of last year. Nor does he think the digestion under pressure should be adopted, on account of the uncertainty introduced by possible variations of pressure when the digestion is carried on in closed vessels. He is of the opinion that the principle of digestion under atmospheric pressure with prevention of evaporation and with definite limits of time, temperature, and strength and amount of acid, is the one best calculated to give uniform results. These conditions obtain in an apparatus similar to that devised by Snyder, of Minnesota, though the condenser is superfluous, as a simple tube of hard glass, 18 to 24 inches long, will answer the same purpose. In regard to the time limit, your reporter considers thirty-six hours unnecessarily long, as giving opportunity for too great decomposition of silicates, and suggests ten hours as a convenient limit. Your reporter desires therefore respectfully to recommend that the above principles be incorporated in any method of soil digestion adopted by this association.

MOISTURE DETERMINATION.

The question of moisture determination is an important one in the analysis of soils, as of the other substances with which we are concerned, for the reason that it is desirable that our results be calculated upon the substance in a uniform and definite condition of dryness. The constant basis adopted by this association in most cases is that of the substance dried at the temperature of boiling water—meaning not 100° C. as determined by the thermometer, but the temperature actually obtained in a vessel surrounded by water kept briskly boiling. There seems to be no reason why the same course should not be followed in the case of soils, though we are met with the statement that some clayey soils continue to lose weight at this temperature for many days, and the reporter of last year adopts the arbitrary procedure of drying for twenty to thirty minutes at 200° in a current of air. The above statement is entirely opposed to your reporter's experience, and is not in accordance with the practice of the late Dr. Robert Peter in his work on Kentucky soils. More than twenty years ago Dr. Robert Peter discontinued the practice of drying soils at 200°, as yielding no certain result, and stated the results of his analyses upon the soil dried in the water oven.

In order to test the matter upon the soils now before this association, your reporter made the following experiments:

Time required for drying.

[Two grams of soil in watch glasses were first exposed twenty-four hours to an atmosphere saturated with moisture, and then dried in a paraffin oven at 195–204° for successive periods of two to four hours, cooling in desiccator and weighing after each period. The results are given in per cent of the air-dried soil.]

Experiment No. 1.								
Number of hours in each period...	3	3	2	3	2	3	4	Total.
Soil sample No. 2, lost...............	4.235	.700	.285	.355	.120	.175	.135	6.005
3, lost...............	3.255	.480	.195	.145	.095	.120	.100	4.390
5, lost...............	2.415	.125	.090	.000	....	....	....	2.630
Experiment No. 2.								
Number of hours in each period...	3	3	2	2	2	3	4	Total.
Soil sample No. 2, lost...............	4.580	.570	.260	.140	.160	.265	.165	6.140
3, lost...............	3.320	.515	.200	.035	....	....	....	4.070
5, lost...............	2.300	.225	.070	.015	....	....	....	2.610

[Two grams of soil were first exposed twenty-four hours to air saturated with moisture, then dried on watch glasses in a water oven kept constantly boiling for successive periods of four hours each, cooling in desiccator after each period. Results are given in per cent.]

	Four-hour periods.				
	First.	Second.	Third.	Fourth.	Total.
Soil sample No. 2................................	2.350	.115	[1].030	[1].030	2.405
3................................	1.835	.110	[1].050	[1].020	1.875
5................................	1.490	.070	[1].020	[1].030	1.510

[1] The soil gained weight in the last two periods. The total indicates the greatest loss observed, diminished by the gain in the third and fourth periods.

The results show that soil No. 2 was still losing weight at 200° even after twenty hours drying, but reached a practically constant weight in the water oven within eight hours. Soil No. 5 is a clayey soil, containing a comparatively small amount of humus. Other trials in which the soil was dried four hours in the water oven, weighed and dried again for periods of two hours, showed that constant weight was obtained in six hours' drying.

It seems reasonable to suppose that soils containing humus, subject to decomposition at high temperatures, and chemical hydrates, which lose water the more rapidly the higher the temperature, but are not completely dehydrated at temperatures short of ignition, would, in the nature of the case, be more likely to reach a constant weight within a reasonable time at 100° than at 200°, and this view is sustained by the figures obtained in the present instance. Dr. de Roode, of West Virginia, in his report, makes the same observation in the case of soils Nos. 2 and 3, and states that he found it impossible to reach constant weight by drying at 200°, but that drying in the water oven was satisfactory. Your reporter, therefore, desires to recommend that the moisture in the air-dried soil sample, as prepared for analysis, be determined by drying a weighed portion to constant weight in the water oven, and that the results of analyses be calculated upon the soil in this dry condition. It is not, of course, claimed that we drive off in this way all the moisture, but we probably do drive off all that is not chemically combined, and we certainly arrive at a nearly constant and definite condition of dryness, which is the end to be attained.

Only two chemists reported on humus, as follows:

Humus determination.

		Thompson.	Jaffa.
		Per cent.	*Per cent.*
Grandeau method	Soil No. 2	1.925	2.43
	Soil No. 3	1.475	1.83
Grandeau method modified by Harry Snyder	Soil No. 2	2.210	....
	Soil No. 3	2.010	....
Huston and McBryde method	Soil No. 2	1.805	....
	Soil No. 3	1.520	....

The determinations by Mr. John Thompson were reported in duplicate, the duplicates being very close in each instance. In the absence of more comparative data it is hardly proper to propose either of these methods for formal adoption by the association, but the modifications of the Grandeau method devised by Huston and McBryde and by Snyder recommend themselves strongly to our attention as being more easily carried out and more accurate in their results than the original Grandeau method.

PHOSPHORIC ACID DETERMINATION.

The very startling variations in the amounts of phosphoric acid reported by different chemists are in the nature of a surprise. It is hardly to be supposed that they are due to differences in the digestion, and as it is to be supposed that the soil solutions obtained by different observers were fairly uniform in their content of P_2O_5, the discrepancies must be due to defects in the method of determination. Your reporter has not had the opportunity to investigate this matter sufficiently. It would seem that higher results are obtained from HNO_3 solutions, but whether these higher results are the correct ones does not yet appear. It will be noticed that the results by the Hilgard method are frequently higher than those obtained by the same observer using the Provisional method. This is a matter that should be thoroughly studied next year.

The question of phosphoric acid remaining in the siliceous residues also needs further investigation. The writer obtained very perceptible amounts of P_2O_5 from the siliceous residues from digestion of soils Nos. 2 and 3 by the Provisional, Hilgard, and Goss methods, the last mentioned being the smallest, amounting to 0.015 and 0.022 per cent of the original soils Nos. 2 and 3, respectively. From the residues of the Provisional methods 0.038 and 0.023 per cent was obtained and from the residues of the Hilgard digestion in HNO_3 quantities varying from 0.016 to 0.086 per cent of the original soil were obtained. It was clearly shown that soil which had been strongly ignited previously to the digestion could not be thoroughly exhausted of its P_2O_5 by two days' digestion in HNO_3, and even after several days in concentrated HCl the residue was still colored with Fe_2O_3 and still contained P_2O_5. It is probable that the P_2O_5 in the residue from the HCl digestion is retained by the humus, and this may afford an argument in favor of destroying the organic matter of the soil by very moderate ignition previous to the determination of P_2O_5, but unless the ignition is very carefully regulated, a more serious error is likely to be introduced by rendering the ferric and aluminic oxides insoluble. It would not be proper to leave this subject without further reference to the method of Goss for rapid determination of P_2O_5.

Besides the results already reported the writer tried this method on some other soils with results as follows:

	Soil sample.			
	No. 5.	2304.	2305.	2306.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*
P_2O_5, Goss method, only precipitated at 65°	0.091	0.354	0.380	0.309
P_2O_5, Goss method, precipitated rapidly	.064			
P_2O_5, by Provisional method	.105	.398	.468	.353
K_2O from the solution prepared as in Goss's method for P_2O_5		.518	.793	.698
K_2O, Provisional method		.513	.723	.630

Mr. Goss obtains in No. 5, 0.086 by his method. The writer obtains better results by precipitating with molybdic solution in the usual manner, but has not yet sufficiently tried the method of rapid precipitation. The method of solution in sulphuric acid is capable of being used for determination of other constituents in soils, and Mr. Goss is now at work in this direction. It is highly probable that a rapid method for P_2O_5, K_2O, CaO, Fe_2O_3, and total N could be devised which would give results entirely comparable with those obtained by thirty-six hours' digestion in HCl. A method of soil analysis consisting in attacking the soil with boiling concentrated sulphuric acid and mercuric oxid, and the determination of iron, phosphoric acid, potash, and nitrogen in the solution, has been proposed by MM. E. Aubin and L. Alla (Encyclopèdie Chimique de M. Fremy, Tome IV (par. A. Müntz) p. 174, Paris, 1888). They set out with the following propositions in regard to the action of boiling H_2SO_4 on soils:

(1) The organic matter, in presence of mercury as in the Kjeldahl method, will be completely destroyed at the end of one hour.

(2) The clay, properly so called, will be completely decomposed at end of two hours.

(3) The calcareous materials will be quickly transformed into sulphate of lime.

(4) The siliceous materials will be slightly attacked at first, and the action of the acid will not increase sensibly in two hours' contact.

Following from these premises they hold that, by causing boiling sulphuric acid to act upon a soil for three hours, they can extract all the elements of fertility that it is capable of furnishing to plants during a long period, and the resources of a soil in elements of fertility can thus be quickly determined.

It is claimed that the method yields much more uniform results in different hands than do the older methods of treatments with HCl or HNO_3.

AVAILABLE P_2O_5 AND K_2O IN SOILS.

Attention is here called to the urgent need of working out methods for determining the availability of plant food in the soil. The recent work of Dyer upon the Rothamsted soils by the use of 1 per cent citric acid, seems to have been in a measure successful.* Your reporter strongly recommends that a study of methods for this purpose on the lines followed by Dehérain and Dyer in Europe and pointed out also by Dr. Kastle in a paper to be presented to this association, be given a prominent place in our work next year. The methods for what may be called the total resources of a soil need perfecting, but we need also methods by which results corresponding to the known results of field experiments may be obtained, and the writer believes that such methods should be based upon a study of the action of dilute vegetable acids or solutions of acid salts, especially oxalates, citrates, and tartrates upon soils of known agricultural capabilities.

SIEVE TO BE USED IN PREPARING THE SAMPLE FOR ANALYSIS.

Your reporter favors the adoption of the ½ mm. sieve as recommended by the reporter of last year. This is in harmony with the system of mechanical analysis of Dr. Hilgard, which has just claims to be regarded as a standard method. The only positive evidence bearing on this question brought out in the work this year is the fact that the iron gravel, or "shot iron ore," removed from soils Nos. 2 and 3 by the ½ mm. sieve was found to contain 1.923 and 2.117 per cent P_2O_5, respectively. As the amount of gravel removed in each case was 2.00 and 4.33 per cent, it appears that the per cent of P_2O_5 in the soil would have been 0.038 and 0.084 per cent higher in each case if the 1 mm. sieve had been used instead of the ½ mm. The per cent of iron would also have been increased. These soils, however, are exceptional in this respect, as in most soils the materials removed would have been mainly siliceous.

MECHANICAL ANALYSIS.

In the absence of any coöperative work it would not be proper to offer a recommendation, but the well-known and carefully-worked-out method of Dr. Hilgard possesses just claims to be regarded as the standard method, while the Beaker method of Osborne presents itself as a suitable Alternate method for those who do not care to undertake the more complicated process of elutriation.

THURSDAY—AFTERNOON SESSION.

The convention resumed its sitting at 1:45 o'clock, and Mr. Peter continued the reading of his report on soil and ash analysis.

Mr. Patterson said that Mr. Nolan, representing the American Fertilizer Company, would like permission to employ a stenographer to take notes of the discussions in the association for the purpose of publishing them in his journal, and suggested that permission be given therefor.

The presiding officer stated that no formal action in such matters was necessary. The meeting was open, and anyone had the privilege of taking notes who desired to do so.

At the conclusion of his report Mr. Peter stated that he had in his possession some papers relating to soil analysis and he would like to call attention to those papers and present an abstract of them and that the papers would be printed in full.

The presiding officer said that Mr. Peter could use his judgment in regard to the parts of the papers which he would present himself and the parts which he would leave for publication.

Mr. Peter then presented the following papers:

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* Journal of the Chemical Society (London), March, 1894.

ON THE ACTION OF OXALIC ACID UPON PHOSPHATES, SILICATES, AND SOIL.

J. H. Kastle, Paul Marvin, J. C. Calvert.

In view of the fact that oxalic acid and its salts occur so widely and abundantly in many kinds of plants, it occurred to one of us (Kastle) that it would not be without interest to study the action of this acid upon mineral substances, particularly those which through their disintegration in the soil furnish some of the elements essential to plant growth.

In view of the rather complex nature of the problem here presented, it was deemed advisable to confine ourselves, for the time being at any rate, to the study of the action of oxalic acid upon phosphates and silicates.

An examination of the literature upon this subject, that is, of such as was at our disposal, revealed that no great amount of work had been done. Two very interesting communications by H. Emmerling[*] are about all that we have been able to find bearing upon this subject, save a very brief reference by Hilgard to the unpublished results of his assistant.

The first communication of Emmerling is devoted to a discussion of the results of his study of the action of dilute oxalic acid upon the nitrates of calcium and the common alkali metals. He showed conclusively that oxalic acid acts upon each of these nitrates with the setting free of nitric acid and the formation of the corresponding oxalates. He concludes, therefore, that free nitric acid must be present in the root sap, and finds this fact a clue to the explanation of the formation of the many various complex nitrogenous compounds within the plant cell.

In his second communication he discusses the results of his study of the action of dilute oxalic acid upon mineral substances, which study he claimed was undertaken with the view to determine the action of the acid upon the insoluble mineral constituents of the soil. He selected calcium carbonate, in the form of Iceland spar, as the substance with which to make the preliminary experiments of this research. The oxalic acid was found to dissolve varying quantities of the spar, depending on the concentration of the acid and physical conditions. It was further found, and this is the most interesting fact observed, that the action of the oxalic acid was very greatly increased by the presence of very small quantities of nitric acid in the solution. For example, he brought upon equal surfaces of Iceland spar 750 cc. of each of the following solutions:

(1) 1–100(N. oxalic acid.
(2) 1–5,000 N. nitric acid.
(3) $\begin{cases}\text{1–100 N. oxalic acid.}\\ \text{1–5,000 N. nitric acid.}\end{cases}$

The acids were allowed to act upon the spar seventy hours. At the end of that time the following quantities of the spar were found dissolved:

(1) 0.0040 gram.
(2) 0.0060 gram.
(3) 0.0240 gram.

In other words, the combined acids dissolved together about 2.4 times as much as did the two acids acting singly.[†] At the close of this second communication Emmerling indicated that the action of oxalic acid would likewise be tried upon phosphates; but so far as we have been able to discover no communication from this or other observers has ever been published bearing upon this subject.

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[*] Ber., **5**, 2, 780, and Ber., **10**, 1, 650.

[†] From a purely chemical standpoint the acceleration produced in the chemical activity of one acid to another is very interesting, and it is our purpose in the near future to make a careful study of this problem.

In fact, judging from the literature, his labors seem to have ended with the investigations which have just been discussed.

ACTION OF 1-10 N. OXALIC ACID UPON SEVERAL VARIETIES OF CALCIUM PHOSPHATES.

As stated above, the wide distribution of oxalic acid in vegetable tissues, in the form of its calcium, and acid potassium salts, naturally suggested that it might be one of the natural agents for the conversion of insoluble into soluble phosphates. Several experiments, therefore, upon natural and artificial phosphates were conducted in the following manner:

A portion of the finely powdered phosphate was mixed in a stoppered flask with the required quantity of a 1-10 N. solution of oxalic acid. The mixture was then allowed to stand for some time, shaking at intervals so as to insure intimate contact between the phosphate and fresh portions of the acid. After standing for the desired length of time, the mixture was filtered upon a dry filter and an aliquot part taken, in which the extent of the decomposition of the phosphate was determined by titrating the oxalic acid remaining in soluble form with a standard solution of permanganate, or by a gravimetric determination of the quantity of phosphoric acid in the solution.

The following are the results obtained:

TABLE NO. 1.—*Action of 1-10 N. oxalic acid on phosphates.*

Number of experiment.	Name of phosphate.	Quantity of calcium in phosphate present.[1]	Quantity of phosphate used in experiment.	Number of cc. 1-10 N. oxalic acid used.	Time.	Number of cc. potassium permanganate used.	Percentage decomposition.
		Per cent.	*Grams.*	*cc.*	*Hours.*	*cc.*	
1	Bone ash	86.57	1	100	75	(2)	13.44
2	Precipitated calcium phosphate	100	1.033	100	72	....	80.7
3	Florida phosphate	36.5	1.2967	100	96	7	93
4	Apatite	91	1.1231	100	96	2	98

[1] Quantity of calcium phosphate in phosphate used.
[2] In experiments 1 and 2 the phosphoric acid was determined gravimetrically.

These results are interesting chemically as showing how greatly a reaction is influenced by differences in solubility of the reacting substances, even though that difference be but small, and they certainly can leave no room for doubt that it is partly by the action of the oxalic acid of the root sap upon the earthy phosphates that these substances, the insoluble phosphates, are converted into soluble form.

ACTION OF 1-10 N. OXALIC ACID UPON THE SILICATES OF CALCIUM AND POTASSIUM.

The ease with which the phosphates were decomposed by oxalic acid naturally suggested that the silicates of calcium, simple and mixed, might also be decomposed by this reagent. In order to determine whether or not such a decomposition could take place, several experiments with different silicates were conducted in much the same way as with the phosphates.

It should be said in this connection that inasmuch as time did not permit a complete analysis of each of the silicates employed, the average composition, as given in Dana's Treatise on Mineralogy, was used in computing the required quantity of the oxalic acid to be employed in each case, and also the percentage of decomposition from the quantity of oxalic acid remaining as such in solution at the end of the experiment. The quantity of oxalic acid usually used in any given case was that required by the quantity of lime, or by the lime and other basic elements of the silicate, except the aluminum present in the amount of mineral used in the experiment. For example:

Chabazite contains, on an average, about 10 per cent of the oxides of calcium, sodium, and potassium together; hence, 16.8 cc. 1–10 N. oxalic acid were used with 1 gram of this mineral.

It was assumed further, in calculating the extent of the decomposition of the mineral from the quantity of the oxalic acid remaining in the solution at the end of the experiment in soluble form, that the formation of calcium oxalate from lime present necessitated a complete breaking down of the mineral molecule.

In each experiment the silicate was ground to a fine powder and introduced into a stoppered flask with the required quantity of 1–10 N. oxalic acid and allowed to stand the desired time, with frequent shaking. At the close of the experiment the mixture was filtered through a dry filter and an aliquot portion titrated with a standard solution of potassium permanganate. The following table is a summary of the results obtained with several silicates:

TABLE NO. 2.—*Action of 1–10 N. oxalic acid on silicates.*

No. of experiment.	Name of silicate.	Composition of silicate.	Quantity of silicate used in experiment.	Number of cc. oxalic acid, 1–10 N. used.	Time.	Number of cc. potassium permanganate used.	Percentage decomposition.
			Gram.	*cc.*	*Hours.*	*cc.*	
1	Pectolite	Ca, Na, SiO_2	1	75	100	19.1	74.66
2	Apophylite	Ca, K, SiO_2	1	44	100	3.96	91
3	Wernerite	Ca, Mg, K, Na, Fe, Al, SiO_2.	1	62	100	51.46	17
4	Wollastonite	Ca, SiO_2	1.16	100	96	18.12	81.88
5	Stilbite	Ca, Na, K, Al, SiO_2	1	13.1	100	10.48	20
6	Chabazite	Ca, Na, K, Al, SiO_2	1	16.8	100	13.44	20
7	Axinite	Ca, Mg, K, Mn, Fe, Al, SiO_2.	1	35.7	100	33.55	6
8	Datolite	Ca, Boro-Silicate	1.6	100	96	11.30	88.7
9	Bytownite	Ca, Na, Mg, Fe, Al, SiO_2.	1	30	100	24.6	18
10	Zoisite	Ca, Mg, Fe, Al, SiO_2	1	40	100	38	5

The filtrate from such of these silicates as contained the one or both of the common alkali metals was tested for these elements with the spectroscope, and in every case one or both were found to be present.

In addition to the experiments tabulated in the above, a few were made with the feldspar, the results of which seem to indicate that this class of mineral is remarkably stable toward oxalic acid, only from 1 to 3 per cent being decomposed. The results, however, obtained in the study of these substances were not altogether satisfactory, and further work is necessary to determine the extent of the decomposition. The extent of the decomposition, however, in the greater number of the cases cited above is certainly remarkably large when we consider the stability usually ascribed to silicates, and in the light of these experiments there can be no doubt that of all the organic acids in the plant sap, oxalic is by far the most powerful in its action upon silicates as well as phosphates. It should be said further that these results have, probably, a still deeper significance when considered in the light of certain conclusions of O. Löw. This observer has shown that oxalic acid and its soluble salts are truly poisonous to the chlorophyl producing plants, and he has reached the conclusion that the poisonous action of the oxalate is due to the decomposition of a calcium compound of the nuclein contained in the cell nuclei and chlorophyll granules, by the oxalic acid or soluble oxalate whereby calcium oxalate is formed. The breaking down of this complex calcium compound causes a destruction of that all-important part of the cell, the nucleus, and a chemical change in the living matter of the cell. Hence the poisonous effect; and hence, inasmuch as oxalic acid seems to be found necessarily within the vegetable cell as one of the products of plant metabolism, it would appear, in the light of Löw's results, that the acid must act almost immediately either upon the calcium salts already present in the tissues of the plant or else upon the calcium minerals of the soil. In other words, there seems to be a physiological as well as a chemical reason, if any distinction can be drawn between them, for the rapid action of oxalic acid upon the calcium minerals of the soil.

Before leaving the discussion of the results above given for the action of oxalic acid upon silicates, it might be well to state that they are given only as closely approximate, for the reason that they are open to two sources of small error. First, the composition of any of these silicates may have departed somewhat from the average composition of the species; and secondly, in the titration with potassium permanganate any sodium or potassium oxalate, which might have been formed in the case of those mixed silicates containing either or both of these metals, would have used up some of this reagent, thereby making the percentage of decomposition too low. Granting both of these sources of error, it may be said that the results above given for the silicates are closely approximate, and quite sufficiently accurate for the support of any theories that have been based upon them.

ACTION OF OXALIC ACID UPON SOIL AT ORDINARY TEMPERATURE.

When this investigation was first undertaken it was the intention to make a comparative study of the action of several representative acids, mineral and organic, upon the soil. Considerable time, however, was consumed in the necessary preliminaries of such an investigation; the mistake was made of taking quantities of soil and acid too small for the subsequent determination of the potash and phosphoric acid liberated, so that finally it became necessary to confine our attention to only one of these acids. Oxalic acid was chosen for much the same reasons that it was used upon those substances.

The several quantities of dilute acid and soil given in Table No. 3 were brought together in large glass-stoppered bottles and the mixture allowed to stand for one month, at ordinary temperature, with frequent shaking. It could easily be seen that action was taking place; the solutions became bright yellow in color, owing to the solution of the iron of the soil, while the specimens of soil lost their original yellowish brown color, becoming gray in color. In the following table (No. 3) will be found a summary of the results of these experiments:

TABLE NO. 3.—*Action of oxalic acid upon soil at ordinary temperature.*

No. of experiment.	Quantity of soil used.	Quantity of oxalic acid.	Time.	Potash taken into solution.	Phosphoric acid taken into solution.	Remarks.
	Grams.		*Month.*	*Per cent.*	*Per cent.*	
1	200	3 L. 1–10 N ...	1	0.06	0.24	By the Provisional method, treating with HCl 36 hours, this soil was found to contain 0.559 per cent potash, 0.473 per cent phosphoric acid.
2	33½	5 L. 1–100 N...	1	.14	.25	

These results are strictly in keeping with the facts already communicated with regard to phosphates and silicates, and certainly go to show that the presence of this acid in the fluids of the plant is an important factor in plant nutrition. It is believed further that these results, aside from theoretical considerations, have their practical bearing as well. First, at the temperature of boiling water oxalic acid acts so rapidly upon the phosphates and silicates of the soil, particularly the former, that it may be that it can be employed successfully in soil analysis in place of the hydrochloric acid method, which is now used provisionally, and which certainly consumes too much time in its operation.

The following experiment looking to this has already been tried, and others are now in progress, the mode of procedure being as follows:

Ten grams of soil are heated in a pressure bottle on the water bath with 100 cc. of N. oxalic acid for six hours. At the end of that time the bottle is opened, its contents filtered, and in aliquot portions of the filtrate the phosphoric acid and potash are determined by the usual methods.

These are some of the results:

TABLE NO. 4.—*Action of oxalic acid upon soils at higher temperatures.*

No. of experiment.	No. of soil.[1]	Quantity of soil used.	Quantity of oxalic acid used.	Time.	Temperature.	Per cent potash obtained.	Per cent phosphoric acid obtained.
		Grams.	*cc. N.*	*Hours.*	° *C.*		
1	No. 1-2500[2]	10	100	6	100	0.436	0.498
2	No. 2-2501	10	100	6	100	.351	.463
3	No. 3-2502	10	100	6	100	.268	.466

[1] Two of these soils, No. 2-2501 and No. 3-2502, were the same samples furnished the association this year by Dr. A. M. Peter.

[2] The soil used in experiment No. 1 was the same as that used in the experiment given in Table 3, and is the same as No. 3, but has received a complete fertilizer every year for six years.

Comparing the results with those of the hydrochloric acid method at present in use, we see that the same quantities of phosphoric acid are removed by the oxalic acid method, with a saving of five-sixths of the time required by the Provisional method.

The potash is not removed as readily for the reason, probably, that by no means all of the potassium exists in the soil in the form of a mixed silicate with calcium, but in other silicates as well. It is hoped that by making use of some other strong acid, such as hydrochloric, in connection with the normal oxalic, it will prove possible to determine the potash as rapidly as the phosphoric acid.

Secondly, the knowledge furnished by these results may find practical application in the way of fertilizing by means of an oxalate. To this there may be possibly two objections. First, the cost of oxalic acid and its soluble salts is at present so great as to preclude its use for this purpose. To offset this, it may be said that certain common plants are known whose roots and tissues contain such quantities of calcium oxalate that, by treatment with sulphuric acid, they might prove efficient manures by supplying not only oxalic acid but nitrogen and gypsum as well; and it should be said further in this connection that our knowledge of the occurrence of the oxalates in plants is by no means complete. There are doubtless a vast number of rapidly-growing, useless plants, weeds, that contain these substances in not inconsiderable quantities; and which, by some such treatment as above suggested, might be transformed from enemies into servants for cultivation.

The second, most apparent, objection which might be urged against the use of these substances as fertilizers grows out of their poisonous nature. It might be urged that the presence of soluble oxalates would poison the growing plant. Such, indeed, would be the case were the oxalic acid or its soluble salts to exist in soluble form in the soil. This, however, could hardly happen, for it has been shown to act so readily upon the mineral matter of the soil that it could scarcely remain in soluble form for any length of time. In fact, it is upon the rapidity of its action that would depend altogether its efficiency as a fertilizer, and by spreading it upon the soil a month or before planting the crop all danger of this kind would be avoided.

At some early date in the future it will be our purpose, conjointly with Dr. A. M. Peter, to make a thorough study, through pot experiments and otherwise, of these several points, believing that the interesting nature of the problem will justify any and all study that may be put upon it.

METHOD FOR THE DETERMINATION OF PHOSPHORIC ACID IN SOILS.

By Arthur Goss.

It is very evident to all who have done work in soil analysis that our present methods, and especially the older ones, consume entirely too much time for practical purposes, and unless they can be very materially shortened, soil analysis must remain of minor importance. The following method is offered as a step in this direction.

Having been thoroughly convinced during a four-years' almost daily experience in the analysis of commercial fertilizers in the Experiment Station of Indiana, during which time I tried nearly all the standard methods for the determination of phosphoric acid, that the Kjeldahl methods were by far the most satisfactory for P_2O_5 in fertilizers, it occurred to me at that time to try to apply the principles involved in these methods to the determination of phosphoric acid in soils. Owing to the lack of time, however, it is only within the last year that I have been able to give the matter attention.

After a large amount of preliminary work, I have succeeded in developing a method which, I think, covers about all the requirements. After using this method in my own laboratory for nearly a year past in the analysis of a considerable number of soils and river sediments, and securing uniformly satisfactory results, I can heartily recommend it as an accurate, quick, direct, and easily manipulated method.

Lack of space will not permit a discussion in this paper of the question of availability. I will only mention the fact that it seems to be generally conceded that plants have the power of ultimately taking all the phosphoric acid out of soils except a small amount, and not, as in the case of potash, being almost totally unable to secure much that may be present. Consequently the question of ultimate availability does not enter into this determination to such an extent as in the case of most others usually made in soils.

In giving the method below, I have gone into quite minute details, as it is well known that the neglect or misapplication of minor points often leads to incorrect results in analytical work.

MANIPULATION.

Weigh out 10 grams of the air-dried soil which has been sifted through a 1-millimeter sieve and transfer to a pear-shaped, straight-necked, Kjeldahl digestion flask, which has been marked to hold 250 cc. (Note 1.) Add approximately 0.7 gram of yellow oxid of mercury and 20 to 30 cc. of concentrated sulphuric acid, as for the determination of nitrogen. (Note 2.) Thoroughly mix contents of flask by shaking; place on a suitable support over a burner; boil for one hour; cool (cold water can not safely be used); add about 100 cc. of water, 5 cc. of concentrated hydrochloric acid, and 2 cc. of concentrated nitric acid; boil gently for two minutes to oxidize iron; cool (cold water can safely be used); make up to volume and filter through a dry folded paper until perfectly clear. (Note 3.) Transfer 100 cc. of the filtrate to an ordinary flask of about 450 cc. capacity, add strong ammonia until a permanent precipitate forms, then 6 or 8 cc. of nitric acid to dissolve precipitate, and boil until clear. (Note 4.) Remove flask from over burner and let cool at the temperature of the laboratory exactly two minutes. (Note 5.) Add 75 cc. of molybdate solution; place the unstoppered flask in an open water bath kept at a temperature of 80° C.; keep neck of flask above water in bath by means of a looped string attached to a support above; keep flask in bath 15 minutes, shaking vigorously four or five times while in bath; remove from bath, let stand 10 minutes to allow precipitate to settle, filter through a 9 cm. No. 589 Schleicher and Schüll paper, or other equally as good, avoiding too strong a pressure at first; wash flask and precipitate thoroughly with ammonium nitrate solution; place flask in which precipitation was made under funnel, shut off pump, and close all valves to filtering jar to form an air cushion and prevent too rapid filtration; fill paper two-thirds full of hot water; add a few cubic centimeters of strong ammonia; aid solution, if necessary, by stirring up precipitate on paper with a small glass rod. (Note 6.) As soon as yellow precipitate is dissolved open valve to filtering jar, but do not turn on pump; after solution has all passed through rinse paper once with a small amount of hot water; after the last portion has passed through remove flask and place a No. 1 lipped beaker under funnel, heat solution in flask to boiling (Note 7); again pour solution through paper, avoiding use of pump at first, otherwise loss from spattering is likely to ensue; wash out flask and paper with a small amount of hot water (the total filtrate should not exceed 50 cc.); add hydrochloric acid to contents of beaker while hot until yellow color appears, then add a few drops of ammonia until solution clears, cool, add 5 cc. of filtered magnesia mixture from a burette, drop at a time, with constant stirring; let stand 15 minutes, add 20 cc. of strong ammonia, specific gravity 0.90; let stand over night (Note 8), filter, wash precipitate with dilute ammonia, dry, ignite intensely over blast lamp for ten minutes, cool in desiccator twenty minutes and weigh $Mg_2P_2O_7$ secured.

Note 1.—Flasks of this description can be had of Eimer & Amend, New York, already graduated, at little additional expense. If such are not at hand, however, it is an easy matter to select and mark some of the small-sized Kjeldahl flasks used for the determination of nitrogen.

Note 2.—Twenty cubic centimeters of acid are nearly always sufficient, but in the case of unusually finely divided clay soils containing little or no sand, it is necessary to use 30 cc. to prevent caking of contents of flask. In doubtful cases 20 cc. of acid should first be added and at the end of five or ten minutes if contents show a tendency to cake, 10 cc. more should be introduced.

Association of Official Agricultural Chemists' samples Nos. 2 and 3 require 30 cc. of acid.

Note 3—In order to secure a clear filtrate it will usually be found necessary to pour the first portion of the filtrate back through the paper three or four times. The filtrate must always be perfectly clear.

Note 4.—In the case of many soils it is not absolutely necessary to oxidize with hydrochloric and nitric acid, as a clear solution can be secured at this point without. In the case of some soils, however, and especially subsoils, the solution can not be cleared up even by prolonged boiling with nitric acid, but if the solution has been previously oxidized, a clear solution can be secured without any difficulty whatever.

Note 5.—In the case of soils containing a large amount of phosphoric acid, the phospho-molybdate forms too rapidly if the molybdate solution be added immediately after removing from over lamp, in which case it is very apt to pass through the paper in filtering. Molybdic acid is also liable to be precipitated. If precipitated as given in directions the precipitate will nearly always be quite granular, giving a clear filtrate in nearly every instance, if good paper is used.

Note 6.—As pointed out by Hilgard, aluminium is sometimes carried down with the phosphoric acid upon precipitating with molybdate solution, in which case some of the phosphoric acid will not be dissolved in the treatment with ammonia. This will be indicated (1) by the appearance of a white precipitate upon dissolving the yellow precipitate in ammonia; and (2) by the difficulty experienced afterward in washing. If such a precipitate be present in any appreciable quantity proceed as follows:

After washing out all the ammoniacal solution in the usual manner, place a small beaker under funnel, close all valves, fill paper one-third full of hot water, add the same amount of concentrated hydrochloric acid, proceed as if dissolving phospho-molybdate in ammonia, receive final solution and washings in flask used for the former precipitation with molybdate solution, add 20 cc. of strong ammonium nitrate solution, neutralize with ammonia, clear up solution with nitric acid, precipitate with 50 cc. of molybdate solution in exactly the same manner as before, filter off phospho-molybdate, dissolve and add to first portion. In case a second precipitation is necessary, it is best to use a No. 2 beaker to receive ammoniacal filtrates.

It is very seldom necessary to make a double precipitation as above, but sometimes it is absolutely essential to do so, no matter how annoying it may be. Nos. 2 and 3 of the present Association of Official Agricultural Chemists' samples furnish the most striking examples of this point that have ever come to my notice.

Note 7.—If solutions have not been oxidized, a blue color is sometimes present upon dissolving the yellow precipitate in ammonia. This can be discharged by boiling the ammoniacal solution for a minute or two and shaking at the same time.

Note 8.—It has been shown that very little precipitate comes down under the above conditions after two hours. I always prefer, however, to let stand over night. If results are required sooner, the filtration can be made at the end of two or three hours and the entire determination made in one day.

The time necessary previous to precipitating with magnesia mixture is usually about three and one-half hours. In case a double precipitation is necessary, about one hour in addition will be required. It is to be understood, of course, that constant attention is not required all this time—for example, the hour's treatment with sulphuric acid requires practically no attention.

REAGENTS.

The reagents employed are the same as recommended in the Association of Official Agricultural Chemists reports under Fertilizers. I give below the method employed in my laboratory for their preparation:

1. Molybdate solution.—Dissolve 100 grams of molybdic acid in 417 cc. of ammonia, specific gravity 0.96; filter the solution thus obtained, not too rapidly and with constant stirring, into 1,250 cc. of nitric acid, specific gravity 1.20, which is kept cool while the ammonium molybdate is being added. Keep in a warm place for three or four days before using.

2. Magnesia mixture.—Dissolve 110 grams of crystallized magnesium chlorid and 280 grams of ammonium chloride in water, add 700 cc. of ammonia, specific gravity 0.96; make up to two liters and filter.

3. Strong ammonium nitrate solution.—Dissolve a weighed amount of ammonium nitrate in a large porcelain dish in the smallest possible amount of water with the aid of heat; make up so that each 20 cc. will contain 15 grams of ammonium nitrate.

4. Ammonium nitrate for washing.—Dissolve 800 grams of ammonium nitrate in water, add 20 cc. of concentrated nitric acid; bring to a volume of 8 liters and filter into a 2-gallon bottle.

5. Dilute ammonia for washing.—One volume of filtered concentrated ammonia to six volumes of distilled water.

In working up this method the following points received attention:

TIME NECESSARY FOR SOLUTION IN SULPHURIC ACID.

The following soils were used for this investigation:

No. 62. A heavy clay loam from the Rio Grande Valley, locally known as an "adobe" soil.

No. 1725. A granular clay soil from post-oak flatwoods, Calloway County, Kentucky.

No. 2131. A clay soil from part of a cultivated field near Beard, Kentucky.

TABLE I.—*Concerning time necessary for solution in sulphuric acid.*

No. of soil.	Hours digested.	Per cent of P_2O_5.
62	½	0.132
62	1½	.132
62	5	.131
1725	½	.054
1725	1	.045
1725	4	.051
2131	½	.059
2131	1	.065
2131	4	.064

By referring to the above table it will be seen that practically no phosphoric acid was dissolved out of these soils after thirty minutes. This, in connection with the fact that I have been able to secure uniformly concordant duplicates in a large number of soils by treating them approximately one hour, has led me to believe that digestion for more than one hour is quite unnecessary.

TIME NECESSARY TO PRECIPITATE PHOSPHO-MOLYBDATE.

The work of the iron chemists has clearly demonstrated that a small amount of phosphoric acid can be completely precipitated in the presence of much iron in so short a time as ten minutes by the use of high temperature, large excess of molybdate solution, and vigorous shaking. It occurred to me that the same principles might be applied to soils. The results obtained with soil No. 62 are given below:

TABLE II.

No. of soil.	Temperature of bath.	cc. molybdate solution.	Minutes in bath.	Minutes to settle.	Per cent P_2O_5.
	°*C.*				
62	80	75	5	15	0.135
62	80	75	5	15	.137
62	80	75	15	15	.137
62	80	75	30	15	.139
62	80	75	1 hour.	15	.142
62	80	75	2 hours.	15	.145
62	80	75	4 hours.	15	.134

From the above it will be seen that practically all the phosphoric acid was thrown down in five minutes. The very slightly higher results in the one and two hour digestions, if they can be attributed to anything except analytical error, and I feel pretty safe in affirming that they were not due to that cause, are probably due to the fact that those precipitates with the molybdate solution contained a slight amount of foreign matter, a trace of which may have been weighed up with the phosphoric acid. The flasks were shaken four or five times while in the bath. It is my opinion that, except to secure a granular precipitate, the shaking is not nearly so essential as the high temperature and large amount of molybdate solution. There is no question in my mind in regard to the completeness of the precipitation when done according to the scheme previously given. I have yet to find a single instance in which there was any evidence to the contrary. The precipitate, when thrown down in this manner is, as a rule, much more granular and easily filtered than when the precipitation is made in beakers in the usual manner.

CONCERNING AMOUNT OF MOLYBDATE SOLUTION NECESSARY.

As is pretty generally known, in the presence of much iron and aluminium, it is necessary to use a large excess of molybdate solution. In the following table are given the results secured on this point. The solution used in each determination represented, as usual, 4 grams of soil.

TABLE III.—*Concerning amount of molybdate solution necessary.*

No. of soil.	Temperature of bath.	Grams Fe_2O_3 and Al_2O_3 in each solution.	Minutes in bath.	Minutes to settle.	cc. molybdate solution.	Appearance of precipitate at end of 20 minutes.	Appearance of precipitate at end of 24 hours.	Per cent of P_2O_5.
	°C.							
62	80	0.5220	15	5	5	None	None	0.000
62	80	.5220	15	5	15	None	Trace........	.000
62	80	.5220	15	5	25	Slight	Slight in filtrate.	.040
62	80	.5220	15	5	35	Normal ...	None in filtrate.	.125
62	80	.5220	15	5	45	....do	....do	.138
62	80	.5220	15	5	55	....do	....do	.136
62	80	.5220	15	5	65	....do	....do	.141
62	80	.5220	15	5	75	....do	....do	.139

From the above it will be seen that in the case of this soil 15 cc. caused no precipitate, that it required between 35 and 45 cc. to cause complete precipitation, and that more than 45 cc. had no effect. This may explain why Hilgard has recommended letting the precipitation with molybdate solution stand "at least twelve hours," as he recommends the use of but 20 cc. of what I understand to be the ordinary molybdate solution. The conditions of course are different, but much iron and aluminium is undoubtedly likely to be present in both cases.

The solutions above contained an amount of oxides of iron and aluminium corresponding to 13.05 per cent. In order to provide for soils containing more of these substances than the above, I have recommended the use of 75 cc. of molybdate solution. This large excess very clearly does no harm, and, in all probability, hastens the precipitation.

CONCERNING THE ADDITION OF AMMONIUM NITRATE BEFORE PRECIPITATING WITH MOLYBDATE SOLUTION.

The precipitations in No. 62, Table I, were made in beakers and stood over night, each receiving 15 grams of ammonium nitrate. The determinations in the same soil, Tables II and III, received no ammonium nitrate, but yielded results very slightly higher.

Taking these facts into consideration, together with the fact that a large amount of ammonium salts is already present, it would seem entirely unnecessary to use ammonium nitrate.

CONCERNING EFFECT OF AN EXCESS OF NITRIC ACID WHEN PRECIPITATING PHOSPHOMOLYBDATE.

The determinations given in Tables I, II, and III each received approximately 6 or 8 cc. of nitric acid to dissolve the precipitate with ammonia when passing from the sulphuric acid to the nitric acid solution before precipitating with molybdate solution. Each of the two determinations given below received 100 cc. of concentrated nitric acid. The solutions and method of precipitation used were the same as that used for the work in Table III.

No of soil.	cc. of nitric acid.	Per cent of P_2O_5.
62	100	0.131
62	100	.134

From this it will be seen that the effect of a heavy excess of nitric acid is extremely slight. The effect of the excess usually employed is in all probability practically nothing.

CONCERNING THE AMOUNT OF PHOSPHORIC ACID LEFT UNDISSOLVED UPON DISSOLVING THE YELLOW PRECIPITATE IN AMMONIA.

The present Association of Official Agricultural Chemists' samples Nos. 2 and 3 were taken for this investigation, as they furnish the best examples of this point that have ever come under my notice. The precipitations were made as described in the scheme, and the residues insoluble in ammonia were treated as described in note 6. The amount of P_2O_5 secured from the residues is given below:

Sample No.	Per cent of P_2O_5.
2	0.053
2	.047
3	.039
3	.036

The second precipitate was almost entirely free from aluminium. The papers, after the treatment with HCl, were carefully tested for phosphoric acid, but failed to show a trace.

From the above it will be seen that in the case of these soils it is absolutely necessary to make the second precipitation. These are very unusual examples, however; in fact, soils containing as these do nearly half a per cent of P_2O_5 are very rare. In nearly all ordinary cases a second precipitation is unnecessary.

COMPARISON WITH OTHER METHODS.

Owing to the large amount of time consumed by the present methods, it is absolutely impossible for one person to make a large number of comparative analyses in any reasonable length of time. Through the kindness of Prof. A. M. Peter, our present reporter on soils, and owing to the fact that I had some samples at hand which I had analyzed eight years before by a different method, I have the following data to present:

Sample No. 1687 is a "virgin soil from crawfish flats of Tennessee River Valley, 2 miles below mouth of Jonathan Creek, Marshall County, Ky."

No. 1690 is a "virgin soil from post oak flats of East Fork of Clarks River, Marshall County, Ky."

No. 1700 is a "virgin soil of the first bottom of Tennessee River, Calloway County, Ky."

No. 1725 has already been described in connection with Table I.

No. 2130 is a "soil from a cultivated field near Beard, Ky."

No. 2131 is "from poorer part of same field as No. 2130."

No. 99 is a cultivated clay soil from Knox County, Ind.

Nos. 123 and 124 are clay subsoils from the same locality as No. 99.

Nos. 1687 and 1725, inclusive, were analyzed by Dr. Robert Peter, for the Kentucky geological survey, by the method extensively used by himself and Hilgard.

Nos. 2130 and 2131 were analyzed by Prof. A. M. Peter, of the Kentucky experiment Station, by the present Provisional method of the Association of Official Agricultural Chemists.

Nos. 99, 123, and 124 were analyzed by myself by the method given in Bulletin No. 10 of the Chemical Division, U. S. Department of Agriculture.

TABLE IV.—*Showing comparison with other methods.*

Number of samples.	By whom analyzed.	Method used.	Per cent of P_2O_5.
1687	Bobt. Peter	Hilgard and Peter	0.136
1687	Arthur Goss	Goss	.114
1690	Robt. Peter	Hilgard and Peter	.113
1690	Arthur Goss	Goss	.101
1700	Robt. Peter	Hilgard and Peter	.218
1700	Arthur Goss	Goss	.264
1725	Robt. Peter	Hilgard and Peter	.076
1725	Arthur Goss	Goss	.050
2130	A. M. Peter	Kedzie	.096
2130	Arthur Goss	Goss	.090
2131	A. M. Peter	Kedzie	.080
2131	Arthur Goss	Goss	.065
99	do	Richards	.076
99	do	Goss	.080
123	do	Richards	.072
123	do	Goss	.086
124	do	Richards	.109
124	do	Goss	.109

The results given above agree quite as closely as could be expected from the method of comparison used. This phase of the work can probably be handled better by sending the same samples to different chemists, as is done through the Association of Official Agricultural Chemists reporters. It may be mentioned here that results of soil analysis are very often reported only to the second decimal place.

The average per cent of P_2O_5 for the nine samples given above, as secured by the method I have proposed, is 0.107; the average given by the other methods is 0.108. This fact would seem to be very suggestive, even admitting the considerable variation in two or three cases. In regard to the close agreement of duplicates secured by this method in individual cases there can be no doubt. I have abundance of evidence on this point in my laboratory notebooks. The results given herewith in connection with No. 62, while hardly comparable as duplicates, still probably present sufficient evidence on this point. It has been my experience that the difference between strictly comparable duplicates, from the start, more often falls within the five thousandth of 1 per cent (0.005 per cent) than in excess of that amount. A difference of anything less than three times the above may be encountered in the case of the present Association of Official Agricultural Chemists' samples Nos. 2 and 3, but should certainly not exceed 0.015 per cent if the determinations are carefully made.

I would respectfully request that the method be tried also on other samples than Nos. 2 and 3, as these are unusually troublesome to work. The method is, however, by far more satisfactory, even with these samples, than any other within my knowledge.

A NOTE ON THE DIRECT DETERMINATION OF POTASH IN THE SOIL SOLUTION.

By Alfred Meredith Peter.

In the usual methods of separating the alkalies in soil analysis, bulky precipitates have to be separated and washed and large amounts of ammonia salts are to be expelled, involving opportunities for serious errors, to say nothing of the accumulation of impurities from the very considerable amounts of reagents that have to be used. In view of these objections it occurred to the writer that it might be possible to separate the small precipitate of $K_2P_2Cl_6$ more accurately by direct treatment, and that by this means a great saving of time might also be accomplished. The experiment was first made on three soils in which K_2O was being determined by the Provisional method. To an aliquot of the HCl solution of the soil enough P_2Cl_4 solution was added to combine with all the potassium chloride present, allowing only a slight excess. The solution was then evaporated to dryness and the residue taken up in the usual way with 85 per cent alcohol and the precipitate filtered on a small asbestos filter, washed as in the Lindo-Gladding method, dried and weighed. The filters were reweighed after washing out the precipitate again with boiling water. The results obtained were distinctly higher than those obtained from the same solutions by the Provisional method, but were close enough to warrant a repetition of the experiment. For the next experiment the solutions obtained from soils Nos. 2, 3, and 5, which were being analyzed for this association, were used. Aliquots representing 1 gram of soil were evaporated with an amount of $PtCl_4$, equivalent to 2 per cent of K_2O or about four times as much as was thought to be present. In this case the beaker was removed from the bath as soon as only about a drop of liquid remained. When perfectly cold, the residues were treated with 85 per cent alcohol as before. The K_2PtCl_6 was in a beautifully crystalline condition. The results, as compared with those obtained by the Provisional method, were as follows:

Per cent of K_2O obtained.

	Soil sample.		
	2	3	5
By Provisional method	0.510	0.480	0.601
By direct precipitation	.458	.433	.566

A repetition of the same experiment on aliquots of the same solution representing 2 grams of soil and using 94 per cent alcohol for taking up the residue, but washing with 85 per cent alcohol as usual, gave slightly better results. In this experiment also, the solutions obtained on washing out the K_2PtCl_6 from the filters with hot water were evaporated with a little more $PtCl_4$, washed and reweighed to ascertain their purity. The following results were obtained:

Per cent of K_2O obtained.

	Soil sample.		
	2	3	5
By Provisional method	0.510	0.480	0.601
By direct precipitation	.482	.446	.586
Same redissolved and reprecipitated	.465	.432	.546

For want of time no more work was done on the method, but it is believed that the subject is worth attention on account of the ease and rapidity with which the determination is made. The K_2PtCl_6 is obtained in good crystals and is readily washed, and seems to be practically pure. The alcoholic filtrate sometimes becomes turbid very soon after filtering, especially if the drying has been carried too far, but this has nothing to do with the results if the filtration is made quickly.

At this moment the Assistant Secretary of Agriculture arrived and was welcomed by the acting president, who expressed his pleasure in greeting in the Assistant Secretary a charter member of the association, and one who had done so much for its usefulness and prosperity. Mr. Dabney said, in replying to the acting president's greeting:

I thank you, gentlemen, for the invitation sent me to be present at your meetings. I certainly feel at home here. I remember well what may be considered the first meeting of this body, which took place at Atlanta in May, 1884. I had the honor to serve as the secretary of that assembly. I look about me now and see new faces. The men who were active at that meeting are represented here to-day by others. Among those who were at Atlanta I recall especially that Nestor of scientific agriculture in this country, Prof. S. W. Johnson. His clear views and well-considered advice did much to give the proceedings their value. This meeting led directly to the one in Philadelphia in September of the same year, in which your organization took its present form. My interest in your success has not in the least diminished since my retirement from active participation in your deliberations, a retirement which has been caused solely by the pressure of other duties. I rejoice at your continued prosperity and usefulness. I am glad you have the Chief of the Chemical Division for your secretary, and hope he may long continue to fill that office. As far as lies in my power, this association shall continue to have the active encouragement and support of the Department of Agriculture. [Applause.]

The presiding officer suggested that in the discussions the members should distinguish between the report itself and the recommendations made therein, and that they should be considered separately.

Mr. WHEELER said, in respect of the content of phosphoric acid in the soil, that according to Hilgard, 0.12 per cent would indicate a sufficient quantity for the nourishment of plants. It all depends, however, upon what percentage of phosphoric acid present can be assimilated by plants, and there is no assurance of the fact that the ordinary methods of analysis give that information. Acetic acid has been suggested by some authorities as a proper solvent to determine availability. It is quite possible to spend a great deal of time and labor to no purpose in long analyses which might be profitably spent if some more definite idea could be secured in regard to the method of determining availability of phosphoric acid.

Mr. PAYNE heartily indorsed the ideas just expressed by Mr. Wheeler. It is important, to be sure, to know the total content of phosphoric acid in a soil, but for practical purposes it is far more important to know what part of it is available.

Mr. LINDSEY said the general tendency of investigation in agricultural laboratories was now in the direction of some methods of determining availability, and he referred especially to the work which Dehérain, Märcker, Lawes and Gilbert, and others had done in this direction.

Mr. PETER desired to call attention to a recommendation he had intended to make, viz, to determine the quantity of phosphoric acid soluble in acetic acid and other reagents of like kind. He thought this would be a good beginning, at least, and then the analyst could gradually work up to the total phosphoric acid. He called attention in this particular to the work done by Dyer and lately published in the proceedings of the English Chemical Society.

Mr. WHEELER asked if he were right in supposing that oxalic acid is as active as hydrochloric acid in dissolving phosphoric acid, and did not Hilgard make this statement?

Mr. PETER remarked that possibly it was more active, but instead of getting available phosphoric acid when we use oxalic acid as a solvent we probably would get the total, or nearly so.

Mr. PAYNE thought it was highly important in all cases to determine available plant food in soil. The plant itself, it must be remembered, produces a certain amount of acidity in the form of organic acids, and this adds very greatly to the solvent action which is carried on in the soil. He thought perhaps that fermentation might be used as one of the means of determining the total available plant food. If organic acids be used as solvents, it must be remembered that they do not always retain their strength, especially if in combination. For instance, he cited citrate of magnesia, which might change its strength and become like a jelly, and thus lose much of its solvent power.

Mr. VAN SLYKE suggested that if there were no further discussion on soil analysis it would be well to proceed to the next subject.

The presiding officer then called for the report on phosphoric acid, which was presented by Mr. Kilgore.

Before Mr. Kilgore read his report the secretary begged leave to read a letter which he had received from Mr. Battle, of the North Carolina Experiment Station.

The presiding officer requested Mr. Battle's letter to be read:

DR. H. W. WILEY,
U. S. Department of Agriculture, Washington, D. C.

DEAR SIR: I regret the fact that I am unable to be present at the meeting of the Association of Official Agricultural Chemists this year, but Mr. B. W. Kilgore will represent the station in my absence.

I beg to call your attention to what I consider a very important matter, which I would suggest should be added as an amendment to the constitution. It is as follows, to be added at the end of section 2:

> No changes shall be made in the methods of fertilizer analysis until an opportunity shall have been given to all institutions exercising official fertilizer control to test the proposed changes.

I speak of this matter now, because at the last meeting in Chicago a change was made in the method for determining phosphoric acid, which was done without careful tests by the different stations, and the result has been to materially reduce the insoluble phosphoric acid. The change was passed quite suddenly, without much discussion, at an adjourned meeting, after the motion was lost the first time. Actions like this I consider very ill advised, and will surely detract from the reputation of the association. I will be glad if you can consider the matter and let it have your support.

Very respectfully,

H. B. BATTLE, *Director.*

REPORT ON PHOSPHORIC ACID.

By B. W. KILGORE.

Early in December last a letter was addressed to various official, commercial, and other chemists soliciting their coöperation in the phosphoric acid work for the Association of Official Agricultural Chemists for 1894, and asking for the suggestion of points deemed worthy of investigation and their methods of carrying out such investigations. Sixty replies were received. After considering these, the literature on recent phosphoric acid work, and the instructions given the reporters at the Chicago meeting of the association, a set of samples was prepared and sent to all chemists (fifty-odd) agreeing to take part in the work, with the following instructions:

INVESTIGATION OF METHODS FOR DETERMINING PHOSPHORIC ACID FOR THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR 1894.

Three samples have been prepared for the coöperative work on phosphoric acid for the present year.

No. 1 is a mixture of the finer portions of cotton-seed meal and castor pomace in approximately equal parts by weight.

No. 2 is an acid phosphate.

No. 3 is a phosphate solution, containing in 50 cc., the equivalent of about 20 per cent P_2O_5 on basis of 0.5 gram substance. This solution was made up at 70° F.

The following plan is outlined for the investigations:

1. The method proposed by Prof. Ross (La. Exp. Sta. Bul. No. 22, pp. 745, 746, and Bul. 38, U. S. Dept. Agriculture, pp. 16, 17) for the "direct determination of citrate-soluble phosphoric acid" in the ammonium citrate filtrate. The method is as follows: "After completion of the thirty minutes' digestion of the sample with citrate solution, filter out at once into a dry vessel 25 cc. of the solution; if the liquid is filtered directly into a dry burette (or measuring flask), 25 cc. can be readily transferred to another vessel without dilution. After cooling, run 25 cc. of the solution into a digestion flask of 250 to 300 cc. capacity, add about 15 cc. of concentrated sulphuric acid, and place the flask on a piece of gauze over a moderately brisk flame. In about eight minutes the contents of the flask commence to darken and foaming begins, but will occasion no trouble if an extremely high or a very low flame are avoided. In about eleven or twelve minutes the foaming ceases and the liquid in the flask appears quite black; about 1 gm. of mercuric oxid or metallic mercury is now added and the digestion continued over a brisk flame. After cooling, the contents are washed into a beaker and the determination continued as under total P_2O_5.

"In case as large an aliquot as 50 cc. of the original filtrate is used, 10 cc. of sulphuric acid are added and the digestion conducted in a flask of 300 to 500 cc. capacity. After the liquid has blackened and the foaming has progressed to a considerable extent, the flask is removed from the flame, 15 cc. more sulphuric acid added, and the flask heated at a moderate temperature for two or three minutes; the mercuric oxid is then added and the operation completed as before described."

It is very necessary in working this method to add the full amount of ammonium nitrate (15 grams.) to the solution before precipitating with molybdate solution, as ammonium phosphomolybdate does not come down readily in the presence of so much ammonium sulphate without the aid of ammonium nitrate.

Should this method prove as satisfactory with others as it has with Prof. Ross, it will simplify and shorten the determination of citrate-soluble phosphoric acid.

To test this method you are requested to determine the citrate-soluble as above described, (*a*) in samples 1 and 2 sent out by the reporter for results on the same samples by different chemists for comparison and (*b*) in any suitable samples you may have on hand, comparing the results with those obtained by the present indirect method of determining the insoluble and calculating the citrate-soluble by difference.

2. The modification proposed by the present reporter (Tech. Bul. No. 7, N. C. Exp. Sta., p. 4) of treating the "citrate-insoluble" residue and filter directly with nitric and hydrochloric acids same as for total in the determination of insoluble instead of igniting. The method is as follows: After digesting with citrate, filtering and washing in the usual way, transfer the filter and insoluble residue to a suitable flask and dissolve in about the same or slightly larger quantities of acids than are used in the nitric and hydrochloric acids method for total. The flasks used for the citrate digestion will be found convenient ones in which to make these solutions, as it will then be unnecessary to remove all the particles from the flasks in washing. The 200 cc. flasks used for making up the solutions may also be used for this, provided ones with sufficiently large necks to take the filters and residue are used.

To test this method you are requested to determine the insoluble (*a*) in samples 1 and 2 by the proposed modification and by the regular Official method of igniting and (*b*) in any convenient samples you may have on hand by the two methods. The insoluble obtained here may be used under 1 for getting the citrate-soluble by difference for comparison with the direct method.

You are also requested to determine the soluble in the above samples, and for No. 1 the water solution will have to be evaporated to dryness and gently ignited.

3. Volumetric determination of P_2O_5 by titrating the yellow precipitate with standard alkali. This method, with different modifications, has been used by a number of chemists during the past few years for determining P_2O_5 in very small quantities as it occurs in iron and steel and their ores. During the past year Mr. Henry Pemberton, jr., has published (Jour. Am. Chem. Soc., Vol. xv, No. 7, p. 382, and Jour. Frank. Inst., 136, p. 362, 1893) the description of a method on this principle which he has applied to the estimation of P_2O_5 in high-grade phosphates. Very satisfactory results have been obtained by this method by a number of chemists, and it is submitted for investigation. The method, as described by Mr. Pemberton, is: "One gram of phosphate rock, or 2 or 3 grams of fertilizer, are dissolved in nitric acid, and without evaporating to dryness diluted to 250 cc. The solution need not be filtered; 25 cc. of the solution are delivered into a four-ounce beaker and neutralized with ammonia until a precipitate just begins to form, and then treated with 5 cc. of nitric acid of specific gravity 1.4, 10 cc. of a saturated solution of ammonium nitrate are added, and the solution diluted to a volume of 50 to 75 cc. It is then brought to a full boil, removed from the lamp, and 5 cc. of the aqueous solution of ammonium molybdate added. This is followed by a second and a third 5 cc., if necessary, the precipitate allowed to settle, and filtered at once through a seven centimeter diameter filter. It is washed thoroughly with water by decantation and on the filter. The filter and precipitate are transferred bodily to the beaker, standard alkali is then run in, and at least 5 cc. of phenolphthalein (1 per cent solution) added, and then standard acid until the color vanishes. Each cubic centimeter of alkali equals 1 milligram of P_2O_5."

The solutions needed are:

(*a*) Ammonium molybdate: 90 grams of ammonium molybdate are dissolved in slightly less than 1,000 cc. of water, allowed to settle, the liquid decanted from the insoluble residue, which latter is dissolved in a small quantity of ammonia and added to the main solution. Any P_2O_5 that may be present is removed by rendering the molybdate slightly alkaline and precipitating with magnesium sulphate. The whole is then made up to 1,000 cc., 1 cc. of which will precipitate 3 milligrams of P_2O_5.

(*b*) A saturated solution of ammonium nitrate.

(*c*) Nitric acid of 1.4 sp. gr.

(*d*) Standard potassium hydrate, 1 cc. of which equals 1 milligram P_2O_5; 100 cc. of this solution will neutralize 32.37 cc. of normal acid, and can be made from normal potassium hydrate (that has been freed from carbonates by barium hydrate) by diluting 323.7 cc. to 1,000 cc.; 1 cc. of this solution equals 1 milligram P_2O_5 (1 per cent P_2O_5 on basis of 0.1 gram substance).

(*e*) Standard acid of same strength as alkali.

(*f*) Alcoholic solution of phenolphthalein, 100 to 1.

Mr. Pemberton used only nitric acid in making his solution, but treats the insoluble residue in the bottom of the flask with a few cubic centimeters of hydrochloric acid and adds to the main solution.

In precipitating, 5 cc. of the molybdate is added and the solution allowed to stand about one minute for the precipitate to settle; then a second 5 cc. is added, if it produces a precipitate, and the solution allowed to stand as before, and so on till no further precipitation takes place. It is never over ten minutes from the time the first molybdate is added till time to filter, and is generally less. The precipitate must be washed thoroughly by decantation, especially if large, and on the filter till the washings no longer have an acid reaction.

The present reporter has made quite a number of determinations of P_2O_5 by the Pemberton method, with fairly good results, though not uniformly good. He has also experimented with several modifications as regards precipitant, precipitation, and filtration, and offers the following as the conditions and form in which the method has given him most satisfactory results for fertilizer work:

Dissolve 2 grams of the substance in 30 to 35 cc. concentrated nitric acid with the addition of a small quantity of hydrochloric (method 4 of the Association of Official Agricultural Chemists, for total), cool, make up to 200 cc., measure out 20 cc. or 40 cc., corresponding to 0.2 and 0.4 gram substance, respectively, into a five-ounce beaker, add ammonia till precipitate just begins to form, and dilute to 60 to 75 cc. If much of the nitric acid was driven off in making the solution, add 10 to 15 grams ammonium nitrate; otherwise this is not necessary. Digest in water bath till temperature of solution has reached about 55° C. (60° C. in open water bath will give practically 55° in beaker) and, after filtering the molybdate solution used in the Association of Official Agricultural Chemists' methods, precipitate in the usual way, allow to stand in the bath five minutes from the time the molybdate is added, remove and filter as quickly as possible upon a filter made by putting a platinum cone or disc, well filled with holes, into a 2½ or 5 inch funnel, and covering with coarse asbestos. By this means a dozen filtrations can be easily completed in ten minutes from the time the molybdate is added to the solution, allowing very little time for molybdic acid to deposit, but sufficient, in my experience, for complete precipitation; but to be sure that all the precipitate is down, put a small amount of molybdate solution into the beaker or pressure bottle placed for the filtrate. Filter paper may be used for catching the precipitate, but I have found less trouble in washing where asbestos was employed. The precipitate is now washed three or four times by decantation, using 50 to 75 cc. water each time and agitating thoroughly, then washed on the filter and till the washings are no longer acid. I test this by emptying the filtrate after having washed with 500 or 600 cc. water, filling the funnel again and adding phenolphthalein and a drop of the standard solution. I have found it to take from 500 to 700 cc. water to free the precipitate of acid. The precipitate is now washed back into the beaker with 25 to 50 cc. of water and titrated as usual.

Where 0.4 gram substance is used for analysis it will be found more convenient to titrate with standard alkali of double the strength recommended by Mr. Pemberton, but using the same strength of acid. I also prefer nitric acid to hydrochloric, as used by him.

You are requested to test this method by both ways of precipitation, and for that purpose please determine total P_2O_5 by the Volumetric method and the regular Molybdate method (*a*) in samples Nos. 1, 2, and 3, and (*b*) in any convenient samples you may have on hand.

It would also be interesting to have results for insoluble in samples Nos. 1 and 2 by the Volumetric method for comparison with the Molybdate—0.5 gram substance is not too much for this purpose.

To get all the total in sample No. 1 it will have to be worked by the magnesium nitrate or ignition method.

In your work on the Volumetric method please report methods of precipitation and filtration, amount of washing, and any other points of interest coming under your observation. It would also be interesting to investigate whether or not so large a quantity of wash water dissolves an appreciable amount of the ammonium phosphomolybdate.

Please report results by the Volumetric method on sample No. 3 in terms of the number of centimeters of your standard alkali required to neutralize the ammonium phosphomolybdate from 20, 40, 50, or any convenient number of centimeters or grams of the solution, and for the results by the Molybdate method on No. 3 give the weight of the $Mg_2P_2O_7$, corresponding to 25 or 50 centimeters, preferably 50 centimeters, or 50 grams.

4. In determining totals by the Molybdate method wash the "white precipitate" (*a*), as is your custom, and (*b*) till free of chlorids, reporting the amount of washing in each case, and results. The reporter would be glad to have any results you may have on hand on this subject, or that you may obtain on samples in your laboratory.

The reporter will also be pleased to receive results or suggestions on any points not asked for in the above outline, especially with reference to testing the neutrality of the "ammonium citrate" and determining insoluble P_2O_5. Full credit will be given for all matter submitted.

All the samples should remain sealed till ready for analysis, and then tightly fitting rubber stoppers should replace the cork ones. Samples 1 and 2 should be well mixed before analyzing them, and No. 3 should be shaken before drawing each portion for analysis.

Should you not have time to investigate all the points outlined, take up those of most interest to you.

A thorough study of one or more of the subjects is much to be preferred to a small amount of work on all. And as two or three samples are not sufficient to test a method, you are requested to use as many samples of different kinds as possible.

It is requested that determinations be made in duplicate at least.

The data obtained will be considered under four heads:

1. Citrate-soluble Phosphoric Acid.

The results of the different analysts for citrate-soluble by the direct method of Ross and the official method by difference on the samples sent out by the reporter are brought together in Table I, and some additional results on other samples are presented in Table II. The results in columns 5 and 6 and in 3 of the two tables, respectively, represent the difference of the direct method from the official, and being greater and less than the results by the Official method.

TABLE I.—*Citrate-soluble phosphoric acid, reporter's samples.*

Name of analyst.	Direct method of Ross.		Official method by difference.		Difference greater (+) and less (—) than Official method.	
	No. 1.	No. 2.	No. 1.	No. 2.	No. 1.	No. 2.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per cent.*	*Per ct.*
B. B. Ross, Alabama Experiment Station	1.59	2.88	1.50	2.95	0.09	—0.07
	1.54	3.29	1.50	3.35	....	....
R. E. Noble, Alabama Experiment Station	1.50	3.28	1.53	3.35	.04	—.09
	1.49	2.25	1.38	3.40	....	....
R. J. Davidson and T. L. Watson, Virginia Experiment Station	1.33	2.16	1.38	2.13	—.02	—.01
	1.37	2.02	1.37	2.08	....	....
E. G. Runyan, U. S. Department of Agriculture	1.10	2.02	1.64	2.19	—.59	—.21
	1.07	1.97	1.68	2.23	....	....
	1.05	2.07	1.66	2.29	....	....
Rudolf de Roode, West Virginia Experiment Station	1.88	2.10	1.43	2.44	.74	—.38
	2.47	2.02	....	....	....	....
T. F. Watson, West Virginia Experiment Station	1.65	1.98	....	....	....	....
	1.83	1.87	1.62	2.19	.07	—.28
	1.60	1.88	....	....	....	....
W. A. Powers, Illinois Agricultural College	1.20	2.44	1.26	2.87	....	....
	1.08	2.64	1.26	2.98	—.14	—.33
H. E. Curtis, Kentucky Experiment Station	1.04	2.77	1.22	3.00	....	....
	1.36	2.28	....	....	....	....
	1.33	2.33	1.18	2.66	.17	—.37
	....	2.30	....	....	....	....
	....	2.26	....	....	....	....
H. B. McDonnell, Maryland Agricultural College	1.60	2.21	1.22	2.28	.38	—.01
	1.60	2.32	....	....	....	....
W. P. Headden, Colorado Experiment Station	1.47	2.57	1.36	2.57	....	....
	1.46	2.57	1.39	2.55	.09	—.03
	....	2.60	1.35	2.69	....	....
R. Harcourt, J. J. Ferguson, P. B. Kennedy, W. McCallum, Ontario Agricultural College	1.58	2.47	....	....	....	....
	1.51	2.54	1.24	....	.29	....
	1.51	2.45	....	....	....	....
H. L. Miller, Wilmington, North Carolina	1.74	1.94	1.71	2.11	.03	—.17
C. B. Williams, North Carolina Experiment Station	1.86	....	1.38	....	.48	....
F. B. Carpenter, North Carolina Experiment Station	....	2.24	....	2.33	....	—.13
	....	2.16	....	....	....	....
B. W. Kilgore, North Carolina Experiment Station	....	2.09	....	2.50	....	—.38
	....	2.14	....	2.52	....	....
	....	2.15	....	....	....	....
John P. Street, New Jersey Experiment Station*	1.92	1.25	....	....	....	....
	1.79	1.25	2.26	1.09	—.40	.16
J. M. McCandless and A. M. Loyd, Atlanta, Ga	2.22	2.85	2.26	2.80	.04	.05
Means	1.52	2.36	1.46	2.60	.06	—.24

* Digested at 40° C.; results not included in averages.

Report received too late to include results in averages:

D. Adriance, Texas Experiment Station	1.61	3.07	1.68	3.06	—0.06	—0.06
	1.62	2.99	1.72	3.12	....	....
	1.67	....	1.67	....	....	....

TABLE II.—*Citrate-soluble phosphoric acid in other samples.*

Analyst and remarks.	Material.	Direct method of Ross.	Official method.	Difference greater (+) and less (—) than Official method.
		Per cent.	*Per cent.*	*Per cent.*
H. B. McDonnell, College Park, Md. (Results are the means of duplicates).	Mixed fertilizer	3.08	3.27	0.19
	do	4.83	5.18	.35
	do	3.20	3.33	.13
	do	2.19	2.31	.12
	do	2.46	2.87	.41
W. A. Powers, Champaign, Ill	Bone superphosphate	7.72	8.22	....
	do	7.58	8.16	....
	do	7.83	8.22	.49
	do	1.23	1.05	....
	do	1.14	1.04	....
	do	1.09	1.05	.10
	do	1.74	1.81	....
	do	1.67	1.76	....
	do	1.66	1.83	.11
H. L. Miller, Wilmington, N. C	Acid phosphate	2.17	2.06	.11
	do	3.52	3.71	.19
	do	1.82	2.05	.23
	do	2.05	2.07	.02
	Mixed fertilizers	3.85	3.61	.21
C. B. Williams, North Carolina Experiment Station.	Acid phosphates	2.56	3.53	.97
	do	2.87	2.78	.09
	do	3.49	4.02	.53
	do	2.81	3.52	.71
	do	3.02	3.20	.18
	do	1.74	2.50	.76
	Mixed fertilizers	1.94	2.80	.86
	do	1.98	2.55	.57
	do	1.66	2.79	1.13
	do	2.16	1.45	.71
	Cotton-seed meal	.93	1.16	+.23
	Castor pomace	1.36	1.44	—.08
Means		2.82	3.08	

COMMENTS OF ANALYSTS ON THE DETERMINATION OF CITRATE-SOLUBLE PHOSPHORIC ACID BY THE ROSS AND OFFICIAL METHOD.

R. E. Noble, Alabama.—The Ross method gives closely agreeing results with the Official method. Ammonium nitrate must be used to aid precipitation. My results were 0.44 per cent low when it was not used.

C. L. Hare.—I am not at all satisfied with my work on the Ross method, and do not report any results.

Wm. P. Headden, Colorado.—I had some trouble with the castor pomace and cotton-seed-meal mixture, and lost several from foaming.

H. B. McDonnell, Maryland.—I am not favorably impressed with the Direct method for reverted. It does not include the P_2O_5 washed out of the sample by the wash water; there is apt to be troublesome foaming when the citrate solution is digested with H_2SO_4, and the time consumed is considerable.

R. de Roode, West Virginia.—The digestion of 25 cc. portions proceeded all right, but none of the 50 cc. ones could be digested to completion on account of uncontrollable foaming. The molybdate precipitate of the 25 cc. portions was a coarse yellow, which formed slowly. After titration and thorough washing, this yellow precipitate was only partially soluble in ammonia, leaving a slimy, greenish yellow residue on the filter, although liberal quantities of ammonia were used. After precipitating with magnesia mixture and standing over night the white precipitate was found to be contaminated with a foreign white, slimy substance. One of these was carried through in the usual way and gave 2.47 per cent citrate-soluble for No. I (as much as the total). Three others were filtered, dissolved in HCl, reprecipitated, allowed to stand over night, and were found to be still some what contaminated and gave 1.88 per cent against 2.47 per cent for No. I and 2.10 and 2.02 for No. II. I have no confidence in these results and consider the method worthless on account of the impurity of the yellow precipitate. I am also of the opinion that with such small quantities of P_2O_5 and such large quantities of sulphates the precipitation with molybdic solution is incomplete. This method has also failed to give satisfaction on various fertilizer mixtures.

H. S. Miller.—I had considerable trouble from foaming and lost several determinations. On the addition of ammonium molybdate there is first formed a white precipitate preceding the formation of the ammonium phosphomolybdate, and on dissolving this latter a greenish yellow residue insoluble in ammonia is left.

C. B. Williams, North Carolina.—Where 50 cc. portions were digested, I had less trouble from foaming when the entire 25 cc. of H_2SO_4 were added at once instead of 15 cc. and later 10 cc. All the molybdate precipitate was not soluble in ammonia, a slimy residue being left on the filter. On precipitating with magnesia mixture and allowing to stand some time, a grayish white precipitate of greater specific gravity than the magnesium ammonium phosphate often settled out, and when this was the case the results were very high.

F. B. Carpenter.—While some good results have been obtained by this method, both on the reporter's samples and others, it is troublesome and uncertain of results. The presence of so much ammonium sulphate retards the precipitation with molybdate, even in the presence of average amount of ammonium nitrate, the precipitation often being incomplete after standing several hours at 65° C.

Reporter.—The Ross method worked very well with me to the point of precipitating with molybdate, where the precipitate was slow to form; the slimy, greenish yellow precipitate insoluble in ammonia, complained of by others, also occurred with me. This residue proved to be a phosphate, but of what I am not prepared to say. A floating flocculent precipitate also formed in the filtrate from the magnesia precipitate after standing over night. This was phosphate of magnesium and ammonia, which had not come down in the regulation time.

DISCUSSION OF RESULTS.

The mean results by this method do not represent anything definite, and differences from mean and difference between highest and lowest results would show still less, since the differences in insolubles would in this way be added to the differences naturally belonging to this method; but the means do show that the Direct method has given slightly higher results on No. 1 and lower ones on No. 2 than the official method, while the differences in Table II on other samples are still more marked in being low. Perhaps the only way to judge of this method on its own merits in comparison with the Official one is to consider the results of each analyst by the two methods separately. From this point of view the results on No. 1 are generally high and low on No. 2, as are also the results on other samples in Table II. The high results on No. 1 would perhaps be accounted for on the basis of the foreign precipitate, but if this precipitate was the same, a white flocculent one, as it was in my case, it was magnesium ammonium phosphate, and belonged to the main precipitate. This flocculent precipitate may not have the same composition as the main one, but

I have seen it several times in the regular method, especially in precipitates from organic materials. It occurred in my insolubles in No. 1, but not in No. 2, and only on long standing (over night). But the precipitate referred to by Dr. de Roode was of a slimy nature, and that by Mr. Williams, of the North Carolina Station laboratory, which came under my observation, was grayish white, considerable in quantity and quite heavy. This occurred in nearly every determination on the reporter's samples, but none of them were carried through, as the precipitates were used in trying to identify the substance. Mr. Williams failed to get any reaction for phosphates. The greenish yellow residue in the molybdate precipitate insoluble in ammonia was undoubtedly a phosphate or held a phosphate, but this should lower the result. The low results on No. 2 and the fertilizers in Table II are either due, we think, to incomplete precipitation by molybdate, and this is very likely since it is well known that ammonium phosphomolybdate does not form readily in the presence of large amounts of ammonium sulphate, especially with the amount of molybdate ordinarily used for precipitation, or else, as suggested by Dr. McDonnell, the citrate digested does not contain the whole of the citrate-soluble phosphate, and that a still further portion is removed in washing with water. In case the former is the true explanation, then the sulphuric acid method for total, and especially the Kjeldahl ones, it seems, would give the same difficulties, as they and the Ross method are the same, the latter having to destroy citrate of ammonia and the former various organic materials; yet, some of the members of the association who condemn in the strongest terms the Ross method for citrate-soluble are the most ardent advocates of the same method for total. Perhaps, then, Dr. McDonnell's explanation is the better one. At any rate, the results by the Direct method for citrate-soluble are, on the whole, not satisfactory.

2. INSOLUBLE PHOSPHORIC ACID.

In Table III are presented the results for citrate-insoluble phosphoric acid on the reporter's samples and also the results by the official ignition and direct solution in acids methods for making the solution of citrate-insoluble. Table IV also contains results on other samples by the two methods of making the solution.

TABLE III.—*Insoluble phosphoric acid, reporter's samples.*

Analyst.	Official ignition method.		Direct solution in HNO_3 and HCl.		Difference, greater (+) less (—) than Official method.	
	No. 1.	No. 2.	No. 1.	No. 2.	No. 1.	No. 2.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
B. B. Ross, Auburn, Ala	0.38	2.70	0.34	....	—0.04	....
R. E. Noble, Auburn, Ala	.27	2.26	.32	2.36	—.02	+0.06
	.32	2.34	.24	2.32	....	....
C. L. Hare, Auburn, Ala	.32	2.26	.30	....	....	....
	.28	....	....	....	....	....
	.34	2.26	.33	[1]2.27	....	....
	.32	2.24	.28	[1]2.30	—.02	+.03
	.34	2.28	....	[1]2.30	....	....
E. G. Runyan, Washington, D. C	....	....	.06	2.97	....	....
	.03	2.73	.08	3.03	+.04	+.23
	.04	2.81	.09	2.99	....	....
R. de Roode, Morgantown, W. Va	.23	3.06	.13	3.13	—.07	+.11
	.20	3.03	.15	3.17	....	....

[1] Volumetric method.

TABLE III.—*Insoluble phosphoric acid, reporter's samples*—Continued.

Analyst.	Official ignition method.		Direct solution in HNO$_3$ and HCl.		Differences, greater (+) less (—) than Official method.	
	No. 1.	No. 2.	No. 1.	No. 2.	No. 1.	No. 2.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
T. F. Watson, Morgantown, W. Va	.19	3.29	.22	3.19	....	....
	.27	3.10	.31	3.25	+.02	+.02
	.22	3.20	....	....	....	....
H. B. McDonnell, College Park, Md	.35	2.99	.24	3.12	−.09	+.13
	.32	3.14	.26	3.28	....	....
Wm. P. Headden, Fort Collins, Colo	.19	....	.18	2.65	....	....
	.19	....	.20	2.60	0	—
R. J. Davidson and T. L. Watson, Blacksburg, Va.	.32	2.95	.27	2.57	....	....
	.27	2.98	.26	2.54	—.02	—.41
R. Harcourt, J. J. Ferguson, P. B. Kennedy, and Wm. McCallum, Guelph, Canada.	.38	3.46	.23	3.34	—.13	+.10
	.31	3.14	.20	3.49	....	....
	.36	3.36	....	....	....	....
W. A. Powers, Champaign, Ill	.45	2.26	.06	2.18	....	....
	.44	2.27	.05	2.26	—.38	—.05
	.40	2.26	.05	2.19	....	....
H. E. Curtis, Lexington, Ky	.36	3.03	.04	2.88	—.27	—.21
	.40	3.07	.18	2.79	....	....
H. L. Miller, Wilmington, N. C	.24	3.29	.25	3.32	+.01	+.03
B. Terne and F. Bergami, Philadelphia, Pa	....	....	....	3.32	....	....
	....	....	....	3.26	....	....
C. B. Williams, Raleigh, N. C	.20	....	.20	2.88	....	....
	.22	2.76	.20	2.90	....	....
	....	....	.20	2.93	—.01	....
F. B. Carpenter, Raleigh, N. C	.21	2.71	.20	2.73	—.01	+.02
B. W. Kilgore, Raleigh, N. C	.20	2.92	.23	2.89	....	....
	.18	2.85	.18	2.81	+.01	—.03
John P. Street, New Brunswick, N. J.[1]	.29	....	....	....	....	....
	.30	4.29	.18	4.26	—.10	—.03
K. P. McElroy, Washington, D. C	.24	....	.23	4.26	....	....
	.26	2.89	.15	....	....	....
John M. McCandless and A. M. Lloyd, Atlanta, Ga.	.15	3.85	....	....	....	....
	.05	....	.09	2.89	....	....
Means	.27	2.82	.19	2.83	—.08	+.01
Greatest variation from mean	.24	.64	.15	.66	....	....
Difference between highest and lowest results.	.42	1.22	.30	1.31	....	....
D. Adriance, College Station, Tex.[2]	.23	3.20	.25	3.24	....	....
	.21	....	.24	....	+.01	+.04
	.25	3.23	.22	3.27	....	....

[1] Digested at 40° C.; results not included in averages.
[2] Report received too late to include results in averages.

TABLE IV.—*Insoluble phosphoric acid in other samples.*

Analyst and remarks.	Material.	Official ignition method.	Direct solution in HNO_3 and HCl.	Difference.
		Per ct.	*Per ct.*	*Per ct.*
A. S. Winton and A. W. Ogden, Connecticut Experiment Station. Molybdate method (digestion method of 1894).	Fish scrap	1.45	1.23	−0.22
	Castor pomace	.04	.01	− .03
	Mixed fertilizers	1.93	1.84	− .09
	do	2.34	2.57	+ .23
	do	.64	.79	+ .15
	do	2.30	2.32	+ .02
A. S. Winton and A. W. Ogden, Connecticut Experiment Station. Citric-acid method (digestion method of 1894).	do	1.23	1.22	− .01
	do	1.48	1.54	+ .06
	do	1.19	1.18	− .01
	do	1.80	1.86	+ .06
	do	1.45	1.49	+.04
	do	1.15	1.09	−.06
	do	1.55	1.46	−.09
	do	1.47	1.49	+.02
	do	3.21	3.18	−.03
	do	1.86	1.86	....
	do	3.07	3.07	....
	do	1.08	.98	−.10
H. B. McDonnell, Maryland Agricultural College. (Results are the means of duplicates. Digestion method of 1894.)	Complete fertilizer	1.82	1.99	+.17
	do	2.28	2.38	+.10
	do	2.36	2.34	−.02
	do	.71	.76	+.05
	do	1.12	1.11	−.01
	Acid phosphate	1.22	1.23	+.01
	do	3.10	3.12	+.02
	Dissolved bone	2.44	2.52	+.08
	do	3.42	3.47	+.05
	Orchilla guano	9.60	9.80	+.20
C. B. Williams, North Carolina Experiment Station. (Digestion method of 1894.)	Castor pomace	.08	.09	+.01
	Mixed fertilizer	2.72	2.72	....
	do	2.21	2.17	−.04
	do	3.76	3.76	....
	do	.77	.84	+.07
	do	.01	.04	+.03
	do	.70	.63	−.07
	do	.33	.33	....
	do	.84	.86	+.02
	do	.56	.51	−.05
	do	2.93	2.92	−.01
F. B. Carpenter, North Carolina Experiment Station. (Digestion method of 1894.)	do	3.72	3.66	−.06
	do	3.28	3.15	−.13
	do	3.83	3.74	−.09
	do	2.23	2.34	+.11
	do	2.74	2.74	....

TABLE IV.—*Insoluble phosphoric acid in other samples*—Continued.

Analyst and remarks.	Material.	Official ignition method.	Direct solution in HNO_3 and HCl.	Difference.
		Per ct.	*Per ct.*	*Per ct.*
B. W. Kilgore and R. E. Noble, North Carolina Experiment Station. (Digestion method of 1893.)	Fish scrap	4.28	4.31	+.03
	......do	4.25	4.22	—.03
	......do	3.99	3.91	—.08
	......do	3.91	3.86	—.05
	Tankage	6.88	6.75	—.13
	......do	6.78	6.72	—.06
	......do	5.71	5.66	+.05
	......do	5.72	5.70	—.02
	Beef azotin	2.88	2.79	—.09
	......do	2.75	2.84	+.09
	Cork azotin	.22	.23	+.01
	......do	.17	.18	+.01
	Dried blood	.41	.32	—.09
	......do	.31	.19	—.12
	Cotton-seed meal	.20	.18	—.02
	......do	.13	.15	+.02
	......do	.13	.15	+.02
	......do	.15	.20	+.05
Means		2.21	2.21	

DISCUSSIONS OF RESULTS.

The work on insoluble shows that there are still wide variations in the results of different analysts despite the statement of last year's reporter that the "discrepancies among chemists in their results on citrate-insoluble phosphoric acid" were due to different working conditions and that the alteration in the method made at the last meeting of the association eliminated "all of the sources of the discrepancies" then so commonly met with. If this really is the case we must look to the personal factor in explanation of the variations in results in this year's work. The results of each analyst, however, agree with each other remarkably well, and the workers in the same laboratories here, as a rule, check well; so it would seem that there are still differences in working conditions, influence of personal factor and likely other causes combining to bring about differences. The variations between the highest and lowest results on last year's sample (mixed fertilizers) by the old method was 1.08 per cent and by the new method 0.36 per cent, the old method giving the higher results; the difference in this year's samples (acid phosphate) by the modified method is 1.22 per cent. These variations, while not as great as some in the past year's, are too great to be overlooked.

With reference to modification in the citrate-insoluble method adopted last year, there can be no doubt that lower results are obtained by it, lower by from 0.20 per cent to even more than 1 per cent, and especially low on acid phosphates. The chief cause of this is to be found in the longer time of digestion at an increased temperature of about 2° C. brought about by the modification; for the temperature of the water in a bath standing at 65° C. in the old method must now be raised to 67° or 68° to give 65° in an open flask, and 65° in an open flask means practically 66° in the closed one as used in digestion.

3. IGNITION AND DIRECT SOLUTION IN ACIDS METHODS FOR INSOLUBLE SOLUTION.

COMMENTS OF ANALYSTS.

R. E. Noble, Alabama.—I find that direct solution in HNO_3 and HCl gives results equally as good as the Official ignition method. Direct solution of the insoluble residue due in acids has been in use in this laboratory for some time and has given satisfaction.

C. L. Hare.—There is practically no difference in results by Official ignition and Direct solution in acids, the latter being used in this laboratory on all materials.

H. B. McDonnell.—The Direct solution in acids method for insoluble I like very much. Our results are a little higher by the new method, due, no doubt, to a slight mechanical loss during ignition and transferring in the one case and to the added bulk of the filter paper in the other.

F. B. Carpenter, North Carolina.—The results by the Official ignition and Direct solution in acids methods give practically the same results, and the latter is shorter and easier of manipulation.

Reporter.—The method of direct solution in acids has been in use in this laboratory for at least five years, "during which time numerous comparisons of it with the Official method upon all the fertilizer materials and mixed goods on our market have been made, and we have yet to find either a mixed fertilizer or fertilizer material that will give results materially different by the two methods."

DISCUSSION OF RESULTS.

The two methods of making the solution of the citrate insoluble have given practically the same results in the reporter's samples, with three exceptions in each case. The results in Table IV on other samples are good, with two exceptions; out of 62 determinations on solutions by the two methods 9 varied by slightly more than 0.1 per cent, and 2 a little more than 0.2 per cent, the averages being the same. Sample No. 1, a mixture of cotton-seed meal and castor pomace sent out by the reporter, was made up as one that would likely give trouble and upon which the method of direct solution in acids would fail if it would upon any material, as these two substances are ones upon which the same method will not work for total. The results are bad from the point of total insoluble contained, but good with three exceptions when each analyst's results are compared on basis of allowable variation. The highest result is 0.45 and the lowest 0.03 per cent, making a variation of 0.42 per cent, twice as much as the total insoluble obtained by the majority of the workers. I am inclined to ascribe these wide variations on No. 1 to the magnesium ammonium phosphate precipitate. A solution containing 0.5 gram substance, with only 0.25 per cent content of phosphoric acid will not, as a rule, precipitate in the same time that a 1 per cent or over 1 will. In my work in No. 1 the "white" precipitates of the insoluble stood three hours alongside those of No. 2 and totals of the same samples, when I obtained 0.08 and 0.09 per cent by the ignition method and 0.06 and 0.12 per cent by direct solution. The filtrates remained overnight, during which time a further precipitate formed in the insoluble filtrates from No. 1, but none in those from No. 2 or totals of either samples. These additional precipitates brought my results up to an average 0.19 per cent for the ignition method and 0.20 for the direct solution one. I have observed in other cases that very small percentages do not precipitate in the same time as larger ones do. I offer this as a possible explanation of the differences on this sample. In operating on cotton-seed meal and castor pomace, I have, as a rule, gotten a few hundredths higher by dissolving the citrate-insoluble residue directly in acids than by ignition, due, perhaps, to the unavoidable small loss in incinerating and transferring. The phosphate in these materials not immediately precipitable by molybdic solution seems to be dissolved by ammonium citrate.

The method of direct solution in acids does not give so nice a solution with organic materials as does the ignition one, but the saving in time where much work is being carried on is not inconsiderable. The direct solution in acids method may be described as follows: After washing is complete transfer the filter with citrate-insoluble residue back to the digestion flasks and dissolve in 30 to 35 cc. HNO_3 and 5 to 10 cc. HCl, boiling forty-five to sixty minutes.

4. Total Phosphoric Acid.

The results for total phosphoric acid, by the Official gravimetric method and by the Volumetric method, as carried out by Pemberton and as modified by the reporter, are brought together in Table V.

DISCUSSION OF RESULTS.

Gravimetric method.—The phosphate solution (No. 3) sent out contained 10 grams c. p. disodium-hydrogen phosphate ($Na_2HPO_4 12H_2O$) to the liter, the theoretical percentage of P_2O_5 in this salt being 19.826. The highest result obtained on this sample was 20.67 per cent and the lowest 19.74 per cent.

TABLE V.—*Total phosphoric, reporter's samples.*

Analyst.	No. 3.					No. 2.					No. 1.				
	Method and washings.					Method and washings.					Method and washings.				
	Official gravimetric.	Volumetric as carried out by Pemberton.	Washings.	Volumetric as carried out by Kilgore.	Washings.	Official gravimetric.	Volumetric as carried out by Pemberton.	Washings.	Volumetric as carried out by Kilgore.	Washings.	Official gravimetric.	Volumetric as carried out by Pemberton.	Washings.	Volumetric as carried out by Kilgore.	Washings.
	Per ct.	*Per ct.*	*cc.*	*Per ct.*	*cc.*	*Per ct.*	*Per ct.*	*cc.*	*Per ct.*	*cc.*	*Per ct.*	*Per ct.*	*cc.*	*Per ct.*	*cc.*
H. B. McDonnell, Maryland Agricultural College.	20.03	20.04		20.20		17.42	16.95		17.03		2.72			2.56	
	20.21	20.04	800	20.20	570	17.39	16.85	800	17.05	625	2.58			2.58	500
B. B. Ross, Alabama Agricultural College						17.69					2.85				
											2.82				
R. E. Noble, Alabama Experiment Station	20.56	20.50	500	20.45	600	17.81	17.79		17.92		2.55				
	20.56					17.86	17.74		17.85		2.53				
	20.65					17.86	17.89	500		600	2.67				
C. L. Hare, Alabama Experiment Station	20.60	20.60				17.88	17.90				2.58	2.55			
	20.61	20.55				17.90	17.85				2.61	2.65			
	20.67	20.20	800			17.81	17.75	600			2.67	2.57	350		
E. G. Runyan, U. S. Department of Agriculture	19.80	20.53	1.020	19.27	1,030	16.82	17.95	1,080	16.94	1,060	2.44	2.48	670	2.58	990
	19.74	20.47	980	19.47	990	16.88	17.78	to	17.04	to	2.50	2.33	840	2.48	to
						16.98		1,120	16.80	1,180	2.49			2.24	1,100
K. P. McElroy, U.S. Department of Agriculture	19.82					17.17					2.42				
	19.87					16.92					2.35				
						17.25					2.26				
R. de Roode, West Virginia Experiment Station	[1] 19.92	20.10		19.75		17.27	17.76				2.51	2.52			
	19.93	19.85				17.09	17.48				2.41	2.44			
	19.87	19.90					17.20				2.46	2.65			
R. J. Davidson and T. L. Watson, Virginia Experiment Station.	20.47	20.00		20.00		17.04	16.99		17.15		2.54	2.49		2.57	
	20.47	19.95		19.85		17.01	17.05		16.99		2.50	2.47	250	2.54	400

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[1] Precipitated with magnesia mixture direct.

TABLE V.—*Total phosphoric, reporter's samples*—Continued.

Analyst.	No. 3.					No. 2.					No. 1.				
	Method and washings.					Method and washings.					Method and washings.				
	Official gravimetric.	Volumetric as carried out by Pemberton.	Washings.	Volumetric as carried out by Kilgore.	Washings.	Official gravimetric.	Volumetric as carried out by Pemberton.	Washings.	Volumetric as carried out by Kilgore.	Washings.	Official gravimetric.	Volumetric as carried out by Pemberton.	Washings.	Volumetric as carried out by Kilgore.	Washings.
	Per ct.	*Per ct.*	*cc.*	*Per ct.*	*cc.*	*Per ct.*	*Per ct.*	*cc.*	*Per ct.*	*cc.*	*Per ct.*	*Per ct.*	*cc.*	*Per ct.*	*cc.*
William P. Headden, California Experiment Station.	20.14			19.95		17.55			17.47		2.43			2.49	410
	20.11			19.94		17.53			17.40		2.43			2.48	to
	20.15			20.10	700	17.60			17.47	800	2.42			2.52	440
T. F. Watson, West Virginia Experiment Station.						17.16	17.30				2.48	2.56			
						17.18	17.36				2.40	2.56			
						17.22	17.42				2.50	2.75			
H. E. Curtis, Kentucky Experiment Station	20.29					17.40					2.52				
	20.32					17.48					2.55				
L. A. Voorhees, New Jersey Experiment Station.	19.99	20.42		20.28		17.18	18.15		17.55		2.62	2.51		2.49	
	19.80	20.54		20.19		17.12	18.00		17.33		2.59	2.51		2.49	
W. A. Powers, Illinois Experiment Station						17.28	16.40	700	16.90	600	2.54	2.30		2.25	600
						17.35	16.25	to	16.70	to	2.59	2.25		2.25	to
						17.40	16.50	800	16.80	700	2.56	2.31	600	2.20	800
R. Harcourt, J. J. Ferguson, P. B. Kennedy, and William McCallum, Ontario Agricultural College.	19.78					17.74					2.23	2.62	500	2.62	500
						17.86	16.69	600	16.69	600	2.21	2.62	600	2.48	600
B. Terne and F. Bergami, Philadelphia, Pa	20.15	20.15		20.20		17.28	17.20		17.15						
	20.28	20.20				17.20	17.15		17.15						
	20.28	20.20					17.25								
H. Pemberton, jr., and J. Hilyard Haines, Philadelphia, Pa.	[1]19.81	19.85	250			[2]17.32	17.14	110				2.38			
	[1]19.80	19.85	300				17.06	200				2.38			

	....	19.85	300	....	....	....	17.28	250	....	....	....	2.44	....	....	....
	....	19.83	340	....	....	....	17.14	150	....	....	....	2.36	75	....	....
	....	19.85	400	....	....	....	17.14	225	....	....	....	....	....	....	....
	....	19.85	500	....	....	....	17.06	200	....	....	....	....	....	....	....
W. M. Allen, North Carolina Experiment Station.	....	....	....	....	....	17.13	....	....	17.15	650	2.52	....	....	2.50	350
	....	....	....	....	....	17.18	....	....	17.00	to	2.52	....	....	2.55	to
	....	....	....	....	....	17.15	....	....	17.10	800	....	....	....	2.55	400
F. B. Carpenter, North Carolina Experiment Station.	[3]19.93	20.00	....	19.85	....	....	....	....	....	....	....	....	....	....	....
	[3]19.94	20.15	....	20.10	....	17.04	17.10	....	17.15	....	2.65	2.58	....	2.55	....
	19.93	....	....	....	....	17.12	17.15	....	17.15	....	2.61	2.63	....	2.58	....
	19.91	....	....	....	....	17.10	....	....	....	....	....	....	....	....	....
B. W. Kilgore, North Carolina Experiment Station.	20.06	20.35	650	19.85	....	17.17	17.20	500	17.15	450	2.42	....	....	2.50	250
	19.93	20.15	850	19.95	650	17.12	17.40	500	17.10	450	....	....	....	2.45	250
	[4]19.83	20.15	850	19.90	850	[1]16.98	....	....	16.97	450	....	....	....	....	....
	[4]19.83	20.10	1,000	19.90	800	[1]16.98	....	....	17.00	500	....	....	....	....	....
	[3]19.91	....	....	19.80	1,000	....	....	....	17.10	500	....	....	....	....	....
	[3]19.85	....	....	....	....	....	....	....	....	....	....	....	....	....	....
C. B. Williams, North Carolina Experiment Station.	[3]20.03	....	....	....	....	....	....	....	17.10	500	....	....	....	2.50	500
	[3]20.05	....	....	....	....	[4]16.96	....	....	17.10	500	....	....	....	2.50	....
Dr. Fassbender[5]	20.03	....	....	....	....	17.32	....	....	....	....	2.40	....	....	....	....
	20.06	....	....	....	....	17.39	....	....	....	....	2.40	....	....	....	....
McCandless and Lloyd, Atlanta, Ga.	20.20	....	....	....	....	17.35	....	....	....	....	2.70	....	....	....	....
Means	20.09	20.14	....	19.96	....	17.31	17.30	....	17.14	....	2.52	2.50	....	2.48	....
Greatest variation from mean	.58	.46	....	.69	....	.59	1.05	....	.78	....	.33	.25	....	.24	....
Difference between highest and lowest results.	.93	.77	....	1.18	....	1.08	1.90	....	1.23	....	.64	.50	....	.38	....
D. Adriance, Texas Experiment Station[6]	....	....	....	....	....	....	....	....	....	....	2.48	....	....	....	....
	....	....	....	....	....	....	....	....	....	....	2.51	....	....	....	....
	....	....	....	....	....	....	....	....	....	....	2.53	....	....	....	....
Dr. Thoms, Riga, Russia[7]	....	....	....	....	....	....	....	....	....	....	2.49	....	....	....	....

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[1] The "white" precipitate was dissolved and reprecipitated.

[2] Contaminated with ferric oxide.

[3] Precipitated with magnesia mixture direct.

[4] One gram citric acid added before precipitating with magnesia mixture.

[5] Citric acid method.

[6] Report received too late to include results in averages.

[7] 21.05 per cent by his method given further on.

The average of all results is 0.26 per cent above the theory, and the greatest variation between the results of all workers is 0.93 per cent. Quite a number of analysts have gotten reasonably close to the theoretical content, a good many have varied widely, and nearly all have gotten high results, showing the tendency of our method to give high results with most chemists, even when the greatest care is exercised, to say nothing of what will be gotten with plain everyday procedure. The variations between the highest and lowest results on samples Nos. 1 and 2 are 1.08 and 0.64 per cent, respectively, which with the variation of 0.93 per cent on No. 3 are, while entirely too wide, less than the differences in any preceding years of which I have found record. Judging from the results on No. 3, where we know the content of P_2O_5, I would say that the highest results on Nos. 1 and 2 are entirely too high, and that those close around 17 and 2.5 per cent represent more nearly the true content of P_2O_5 in these samples. Where does the trouble lie? It will be observed that the results on No. 3 Gravimetric method marked "3," where the analysts precipitated the solution direct with magnesia mixture, are, while slightly high most of them, practically the same as when previous precipitation with molybdate solution was made. This would seem to indicate that the difficulty is not in the molybdate precipitate or precipitation. Further it will be seen that results marked "2," where solution No. 3 was precipitated in the usual way, and after washing the "white" precipitate, dissolving it in HCl, and reprecipitating with ammonia, are very close to the theory; and that those marked "4," where 1 gram citric acid was added just before precipitating with magnesia mixture, are almost exactly theoretical ones. This would seem to indicate that either some of the magnesia of the magnesia mixture is thrown down as hydroxid and contaminates the precipitate, or else the magnesium ammonium phosphate formed under our conditions of precipitation contains more than a normal amount of magnesium, and that by dissolving the "white" precipitate in HCl and reprecipitating, and also by the presence of citric acid in the solution where the white precipitate is formed, the trouble is prevented.

H. Neubauer* claims that magnesium ammonium phosphate formed in solution containing excesses of both ammonia and magnesium salt, which is our condition, contains more than a normal amount of magnesium.

It is just here, I think, that an important error creeps into our results, and it is not at all improbable that this error is greater, the greater the excess of magnesia mixture and the stronger alkaline the solution, though in support of the view of magnesium of the magnesia mixture precipitating as the hydroxid and contaminating the "white" precipitate, if I may be pardoned an allusion to a bit of personal experience, I will add that I had it on one occasion some years ago where very large excesses were used and long standing allowed, fourteen or fifteen hours, to do so to the extent of increasing my results from a few tenths to more than 1 per cent, according to the time of standing. That this was magnesium hydroxid was demonstrated by making a solution corresponding in alkalinity (but of full alkaline strength) and otherwise to the one from which magnesium ammonium phosphate is precipitated and adding to this greatly more than the usual amount of magnesia mixture; after standing some ten or twelve hours a precipitate commenced to form and continued for twenty-four hours or more, when it was quite considerable. This, however, was an extraordinary case, and I do not think that any very considerable error is introduced into a result in this way where too great an excess of magnesia mixture is not used and too long standing allowed. It has been reported more than once, as the result of experiment, that citric acid in the solution from which the magnesium ammonium phosphate is precipitated will prevent its containing excess of magnesia. This was the case in sample No. 3, judging by the results, and the results by the reporter and Mr. Williams on No. 2 are quite concordant and lower where citric acid was used than where it was not. I will say, however, that the precipitate formed in the presence of citric acid is very fine and with us could only be caught by the use of double filters. It is hardly necessary to say that the total P_2O_5 in the castor pomace and cotton-seed meal sample was only gotten in form precipitable by molybdic solution by ignition previous to solution. The following are some results on it by solution in acids direct:

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* Journal Am. Chem. Society, May, 1894, p. 289.

Gravimetric method, solution in HNO_3 and HCl.—B. W. Kilgore.	Volumetric method, solution in HNO_3 and HCl.—B. W. Kilgore.	Volumetric method, solution in (1) HNO_3KClO_3. (2) HNO_3 and HCl.—H. Pemberton.
0.84	0.825	0.78 (1)
....	.80	.78 (1)
....	....	.66 (2)
....	....	.40 (2)
....	....	.42 (2)

RESULTS OF FOREIGN WORK.

The reports of two foreign chemists have been received through the Chemical Division of the U. S. Department of Agriculture, and are inserted in full. The methods used differ from ours, but such results as are comparable with those of American workers appear elsewhere in the tables.

Results Obtained by Dr. Friedrich Strohmer at the Experimental Station of the Central Union for Beet-Sugar Industry in Austro-Hungary.

The following report, sent by Dr. Strohmer, did not come in time for incorporation with the results given by the reporter. The estimation of the phosphoric acid samples Nos. 1 and 2 was conducted as follows:

1. The estimation of the citrate-soluble phosphoric acid was conducted according to the method of the Halle Station, using Petermann's solution. The work was conducted as follows:

Two grams of the substance were digested with 100 cc. of Petermann's solution of specific gravity 1.09 for half an hour at 50° in a beaker. Afterwards it was filtered with the use of a filter pump and washed with a one to one mixture of water and Petermann's solution until the dissolved phosphoric acid was removed completely from the filter. The filter was dried, incinerated, and the residue dissolved with 2 cc. of nitric acid and 20 cc. of sulphuric acid in a 200 cc. flask, filtered, and 100 cc. of the filtrate used for the estimation. This quantity was almost neutralized, and then treated with 50 cc. of citrate solution, 25 cc. of magnesia mixture, and 20 cc. of 24 per cent ammonia. The filtration of the ammonia-magnesium phosphate was not made until after forty eight hours. The difference of the total phosphoric acid and of the part insoluble in citrate of ammonia gave the quantity of citrate-soluble phosphoric acid.

2. The estimation of the total phosphoric acid according to the molybdate method or according to the citrate method.

In both methods the phosphoric acid was brought in solution by the same processes and the determination was made in the following manner:

Five grams of the substance were boiled for half an hour in a half-liter flask with 20 cc. of nitric acid of a specific gravity of 1.42 and 50 cc. of pure concentrated sulphuric acid. Afterwards the contents of the flask were cooled and filled to the mark at 17.5°, thoroughly shaken and filtered.

(*a*) Of the filtrate for the molybdate method, 50 cc. were treated with the molybdate solution and the analysis was finished according to the method of Sonnenschein. (*See* Fresenius, 6th German edition, 1875, vol. 1, page 404.)

(*b*) In the citrate method 50 cc. of the filtrate were added to 100 cc. of the citrate solution and afterwards, drop by drop with constant stirring, 25 cc. of the magnesia mixture added. The preparation of the two solutions is described in the pamphlet, Agriculturversuchsstation Halle, by Drs. Bieler and Schneidewind, on page 62.

3. *Estimation of the water-soluble phosphoric acid.*—Ten grams of the dried substance were brought into a 1-liter flask and 800 cc. of water added. The flask was shaken for two hours, filled up to the mark, shaken again vigorously, and filtered. Of the filtrate, 100 cc. were treated either by the Molybdate method or according to the citrate method.

4. The estimation of the citrate-soluble phosphoric acid, according to Ross, and the estimation of the citrate-soluble phosphoric acid by difference were determined by the method sent. The determination of the phosphoric acid by the Volumetric method of Pemberton was not tried.

Table of results.

	Sample No. 1.		Sample No. 2.	
	Molybdate method.	Citrate method.	Molybdate method.	Citrate method.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Citrate-soluble phosphoric acid	....	0.00	....	1.86
				1.83
Total phosphoric acid	2.53	2.50	15.73	15.80
	2.51	2.55	15.79	15.75
Soluble phosphoric acid	0.00	0.00	12.08	12.03
			12.05	12.05
Citrate-soluble phosphoric acid according to Ross	1.50	Not done	1.63	Not done.
	1.31		1.49	
Citrate-soluble phosphoric acid by difference	1.41	1.35	1.54	1.82
	1.44	1.32	1.65	1.75

In regard to these results it is noted that the estimation of the citrate-soluble phosphoric acid, according to the Halle method, as well also as the estimation of the total phosphoric acid and soluble phosphoric acid, is much more quickly obtained than the estimation of the citrate soluble phosphoric acid according to Ross and the estimation of citrate-soluble phosphoric acid by difference.

The soluble phosphoric acid in sample 3 of the phosphate solution was determined in 50 cc. by the molybdate and citrate methods under the same conditions as have been given above. The results were as follows:

Molybdate method.	Citrate method.
0.0998 gram P_2O_5.	0.1008 gram P_2O_5.
.0995 gram P_2O_5.	.1001 gram P_2O_5.

For 100 cc. the figures would be double the above.

REPORT BY DR. FASSBENDER.

PHOSPHORIC ACID IN SAMPLE NO. 1.

Total.—Ten grams of substance were boiled in a flask with 100 grams of sulphuric acid till the fluid was clear and colorless. After cooling it was diluted and made up to 500 cc. A 100 cc. portion was measured and to it added 20 cc. of citric-acid solution (500 grams citric acid to a liter of water) and then 70 cc. of 10 per cent ammonia.

Before the fluid became cold 25 cc. of magnesia mixture were added, and the bottom of the beaker lightly rubbed with a rubber-shod glass rod. When a cloud of the phosphate was seen to form, the rod was withdrawn and the adhering solution washed back with a little diluted citric acid. It was allowed to stand two or three hours when the precipitate had completely settled. It was then filtered, washed with 2.5 per cent ammonia, and ignited till complete whiteness was attained. Results obtained were:

$Mg_2P_2O_7$.	P_2O_5.
Gram.	*Per cent.*
0.0751	2.40
.0754	2.41

PHOSPHORIC ACID IN SAMPLE No. 2.

Water-soluble phosphoric acid.—Ten grams were placed in a 500 cc. flask, the flask a third filled with water, and then shaken half an hour. It was filled up to mark, the liquid filtered, and 25 cc. taken for estimation of P_2O_5 in the way already described.

Citrate-soluble phosphoric acid.—Two grams of substance were rubbed up in a dish with 100 cc. of Petermann's citrate solution, brought into a 250 cc. flask and digested one hour at 40°. The solution was made up to the mark and filtered. One hundred cubic centimeters of the filtrate were taken for the determination of phosphoric acid, which was effected as before given. Petermann's solution is made by dissolving citric acid in ammonia till neutrality is obtained, bringing the solution to 1.09 sp. gr. and then adding 50 cc. of ammonia to each liter.

Total phosphoric acid.—Five grams of substance were heated with aqua regia in a 500 cc. flask placed on the water bath. After cooling, the liquid was filled up to the mark, filtered, and 50 cc. taken for phosphoric acid determination.

	$Mg_2P_2O_7$.	P_2O_5.
	Gram.	*Per cent.*
Water-soluble	0.0930	11.90
	.0932	11.93
Citrate-soluble	.0800	12.80
	.0801	12.82
Total	.1353	17.32
	.1359	17.39

PHOSPHORIC ACID IN SAMPLE No. 3.

Twenty-five cubic centimeters of the solution containing phosphoric acid sent out as sample No. 3 were measured out and the phosphoric acid determined as before described.

	$Mg_2P_2O_7$.	P_2O_5.
	Gram.	*Per cent.*
In 25 cc	0.0784	20.06
	.0783	20.03

PHOSPHORIC ACID METHODS USED BY PROF. G. THOMS, OF RIGA.

In the case of No. 1 (mixed castor pomace and cotton-seed meal) solution was effected by the method used at the Riga Station for soils. A portion was burnt, the ash dissolved in strong hydrochloric acid, the solution evaporated to dryness, the residue moistened with nitric acid, then taken up with water, and the solution brought to volume. A 25 cc. portion (in this case representing 1.4408 grams of substance) was mixed with 75 cc. of molybdate solution, each cubic centimeter of which contained 43 milligrams of molybdic acid. The mixture was allowed to stand twelve hours on the top of a steam bath, though not in direct contact with steam. It was then filtered and the precipitate washed with ammonium nitrate solution prepared by mixing 3 kilos of ammonium nitrate, 2 liters of 1.2 sp. gr. nitric acid and 15 liters of water. The precipitate was dissolved on the unperforated filter by dilute ammonia (1 : 3), the solution mixed with 10 cc. of magnesia mixture prepared with magnesium chlorid and allowed to stand over night. The double phosphate was converted into pyrophosphate over a Bunsen lamp and then ignited to a white heat before the blast lamp for five to ten minutes, or sufficient to bring it to whiteness and constant weight. The per cent found was 2.49.

In sample No. 2 (acid phosphate) only soluble phosphoric acid was determined. Twenty grams were transferred to a liter cylinder and water added to the mark. The mixture was then shaken every ten minutes for two hours, the time of shaking being regulated by a sand glass. Liter flasks are not used for this determination at the Riga Station, it being found easier to intermix water and solid substance in a cylinder. The solution was then filtered through a dry filter and 10 cc. taken for the determination of phosphoric acid. The estimation was conducted as before described. The per cent found was 13.53.

No. 3 (a solution) was precipitated with molybdate solution, a 10 cc. portion being taken. The rest of the determination was as before described. The result of the determination gave a mean of 0.2105 gram of P_2O_5 to the 100 cc.

VOLUMETRIC METHOD FOR TOTAL PHOSPHORIC ACID

Comments of Analysts on Volumetric Method

R. E. Noble, Alabama.—Neither of the Volumetric methods would give results that could be relied on on the solutions from the Ross method of digesting for citrate-soluble on account of the deposit of molybdic acid. This deposit did not take place in solutions by other methods. The Volumetric method as carried out by Pemberton and Kilgore gives results agreeing with the Gravimetric method. The results by the Volumetric method are on the whole good, but there is a lack of uniformity, which I can only account for by the deposit of traces of molybdic acid or the excessive washing dissolving some of the yellow precipitate, or both. I found that traces of the yellow precipitate were dissolved when the washings became nearly neutral, but reprecipitated on following into the acid filtrate. Insolubles by the Volumetric methods were uniformly good, agreeing with the Gravimetric results in every case. For total, 0.5 gram is too much; when 0.2 gram was used the results were more uniform and required less washing to free the precipitates of acid.

C. L. Hare.—In citrate-soluble by the Ross method and total by the sulphuric-acid methods precipitation by aqueous solution of ammonium molybdate is not ready and requires some minutes for completion, owing to the large amount of ammonium sulphate. The large amount of washing is the weak point in this method. In a 20 per cent material 0.2 gram is the most convenient portion for analysis.

H. W. Wiley.—In the Pemberton method, and also in Kilgore's modification, the results are very high, but such good results have been obtained with it by other analysts that I am not yet ready to condemn it as being unreliable on a single trial. The trouble appears to be that in precipitating the hot mixture some molybdic acid is thrown down and this affects the titration.

W. A. Powers, Illinois.—I found the yellow precipitate slightly soluble in water. After filtering by decantation and washing on filter to 500 cc., the next 100 cc. or more were evaporated to dryness, when quite a yellow residue was left.

H. B. McDonnell, Maryland.—I fail to see any advantage for the Pemberton method in official work. The protracted washing of the yellow precipitate needed to remove the acidity offsets any advantage it might otherwise have as to time. The end reactions are frequently not as distinct as desirable. I prefer the reporter's modification.

R. de Roode, West Virginia.—I do not believe that water dissolves the yellow precipitate, but water alone used for washing allows the precipitate to pass through the filter, especially at the end, when all acid and ammonium nitrate are washed out. I find that if the main washing is performed with 2 per cent ammonium nitrate solution, and finally, washed three or four times with water, better results are obtained. The results obtained when small quantities of the yellow precipitate passed through were, as a rule, about correct, whereas those where the filtrate was perfectly clear were too high, and I am inclined to believe that the alkali is too weak and that alkali of the former strength of 326.5 cc. normal alkali to the liter is more nearly correct than that of 323.7 cc.

A. E. Shuttleworth, Canada.—The Volumetric method is very much quicker and less tedious than the Official method, and our results by the Pemberton and Kilgore methods compare very closely.

H. Pemberton, jr.—The yellow precipitate was in all cases filtered upon a 2-inch Hirsch funnel (a porcelain funnel with perforated porcelain plate cast in it), whose perforated plate was covered with a disk of soft filter paper, such as comes in cut filters, as it shapes itself to the filter better than the sheet paper. Practically 0.2 gram substance was used for analysis; 250 cc. wash water freed No. 3 of acid, and additional washing of 250 cc. did not lower results.

C. B. Williams, North Carolina.—My results by the Volumetric method were generally very good, but tended to be a little high. My later results by this method, as carried out by the reporters, have all been good.

F. B. Carpenter.—On all low percentages the results by the Volumetric method, as carried out by both Pemberton and Kilgore, compare well with the Gravimetric method, but on higher percentages the results were slightly higher. The best results were obtained by using the Gooch crucible and pump for filtering.

Reporter.—The Volumetric method, as carried out by Pemberton, has given me, on the reporter's sample, fairly good results; as carried out by myself the results compare well with the Gravimetric, but the results on all other samples operated on have not been just what would be liked by either method of procedure. On small percentages no trouble has been had; 0.2 gram substance is best for percentages above 4 or 5, but for lower ones 0.4 gram can be used to advantage. The easiest method of filtration with me has been the use of a 3-inch funnel with platinum cone or disk well filled with holes and covered with coarse asbestos, using filter pump. This funnel will take the entire filtrate from a molybdate precipitate at once, which runs through quickly, allowing but little time for molybdic acid to deposit, and can be washed easily with large quantities of water at a time. The Hirsch funnel has proved very satisfactory, but the filtration and washing are more tedious on account of the shallowness of the funnel and liability of spurting out some of the precipitate in stirring it up with wash water. A deep funnel of the same kind would be most excellent, and I think the new porcelain disk with rubber rim will be a good substitute. The yellow precipitate is slightly soluble in water. I found 1,000 cc. wash water to dissolve an amount of yellow precipitate corresponding to about 0.15 per cent P_2O_5 on basis of 0.5 gram substance.

DISCUSSION OF RESULTS.

The results for total phosphoric acid by the Volumetric method are presented in Table V, besides those by the Gravimetric, and also the amount of wash water required to free of acids, when reported. The results reported are decidedly encouraging in comparison with those by the Official method. On Nos. 2 and 3 the variations between highest and lowest results by the Volumetric methods are wider than those by the Official method, but the averages by the Volumetric method, as carried out by Pemberton, are practically the same as by the Gravimetric, while the averages by the Volumetric method, as described by the reporter, represent more nearly what we believe to be the phosphoric-acid content of the samples. On No. 1 the Volumetric results are good, and the variations between results and from averages are less than the Gravimetric ones. It is not believed, however, from the comments of analysts that the Volumetric methods will at present give results on high percentages to be relied on invariably, and are not so sure as the official one.

The results for water-soluble phosphoric on Nos. 1 and 2 are presented in Table VI. The water soluble in No. 1 was not all directly precipitable by molybdic solution, and had to be evaporated to dryness and ignited.

TABLE VI.—*Water soluble phosphoric acid, reporter's samples.*

Analyst.	No. 1. Method and washings.			No. 2. Method and washings.				
	Official gravimetric, evaporated and ignited.	Direct precipitation of water extract.	Volumetric as carried out by Kilgore.	Official gravimetric.	Volumetric as carried out by Pemberton.	Washings.	Volumetric as carried out by Kilgore.	Washings.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*cc.*	*Per ct.*	*cc.*
R. E. Noble	0.73			12.20				
	.73			12.17				
	.97			12.20				
B. B. Ross	.97			12.04				
John P. Street		0.08		11.80				
		.14		11.72				
		.12		11.84				
C. L. Hare	1.09			12.13				
	1.13			12.14				
	1.11			12.14				
E. G. Runyan	.74			11.66	12.75		12.30	
K. P. McElroy				11.69			12.65	
R. de Roode	.82	.55		11.91				
	.86	.52		11.86				
R. J. Davidson and T. L. Watson	.84			11.96				
	.86			11.95				
T. F. Watson	.67			11.78				
	.64	.43		11.85				
	.82	.45		11.81				
H. E. Curtis	.99			11.81				
	.97			11.64				
R. Harcourt, J. J. Ferguson, P. B. Kennedy, and Wm. McCallum.	1.07			11.66				
	1.05			11.70				
	1.17			11.80				
C. B. Williams	.84	.25	.90	11.97	12.00	18 times.	12.00	15 times.
	.83	.25	.87	12	12.25		12.05	
	.84		.85	12.08	12.20		11.15	
F. B. Carpenter	.73		.80	12.08	12.20		12.12	
	.78			11.99	12.00			
B. W. Kilgore				11.80			11.85	450
				11.74			11.75	
Dr. Fassbender[1]				11.90				
				11.93				
McCandless and Lloyd		.44		11.70				
Means	.89	.32	.86	11.90	12.25		12.11	

[1] Citric-acid method.

In Table VII are brought together those results on insoluble phosphoric acid made by Gravimetric and Volumetric methods. The comparisons are good, with one or two exceptions, and we consider the Volumetric method safe for such low percentages when properly understood and worked.

TABLE VII.—*Insoluble phosphoric acid, reporter's samples.*

Analyst.	No. 1. Method and washings.					No. 2. Method and washings.				
	Official gravimetric.	Volumetric as carried out by Pemberton.	Washings.	Volumetric as carried out by Kilgore.	Washings.	Official gravimetric.	Volumetric as carried out by Pemberton.	Washings.	Volumetric as carried out by Kilgore.	Washings.
	Per ct.	*Per ct.*	*cc.*	*Per ct.*	*cc.*	*Per ct.*	*Per ct.*	*cc.*	*Per ct.*	*cc.*
R. E. Noble	....	....	....	....	....	2.26	2.20	....	2.28	....
	....	....	....	....	....	2.34	2.32	....	2.32	....
	....	....	....	....	....	2.26	2.26	....	....	....
C. L. Hare	....	....	....	....	....	2.24	2.27	....	....	....
	....	....	....	....	....	2.26	2.30	....	....	....
	....	....	....	....	....	2.28	2.30	....	....	....
E. G. Runyan	....	....	....	....	....	2.73	3.20	....	....	....
	....	....	....	....	....	2.81	3.38	....	....	....
R. J. Davidson and T. L. Watson	0.32	0.20	....	0.15	....	2.95	2.82	....	2.84	....
	.27	.20	250	.15	400	2.98	2.77	250	2.90	400
B. Terne and Bergami	....	....	....	....	....	3.32	3.25	....	....	....
	....	....	....	....	....	3.26	3.20	....	....	....
C. B. Williams	.20	....	....	....	....	2.90	....	....	2.87	....
	.20	.15	....	....	....	2.93	3.10	....	2.87	350
F. B. Carpenter	.21	....	....	....	....	2.71	....	....	....	....
	.25	....	....	.21	....	2.76	2.90	....	2.80	....
B. W. Kilgore	.20	....	....	.20	....	2.92	....	....	2.90	250
	.18	....	....	.18	....	2.85	....	....	2.75	250
	.23	....	....	.23	....	2.89	....	....	2.85	250
	.18	....	....	.18	....	2.81	....	....	2.80	250
Means	.224	.18	....	.19	....	2.72	2.73	....	2.74	....

With reference to the Volumetric method as carried out by H. Pemberton, and as described by the reporter, there are two main differences: First, the temperature of precipitation in the former is at boiling; in the latter 55° to 60° C.; 60° in both gives practically 50° in beaker. At the former temperature molybdic acid deposits very quickly, especially if any considerable excess of the precipitant is present. If we know how much phosphoric acid our samples contain and add just enough molybdate to do the work, or are willing to add little by little of the precipitating reagent till the proper amount has been added, this objection is largely overcome, but the former we do not know and the latter is tedious and time-consuming. At 55° C. the official molybdate does not deposit for fifteen minutes or more at most, precipitation is always complete in 4 or 5 minutes from the time the molybdate is added, and precipitation and filtration can be finished in almost, if not in as short a time by this method of procedure as by the regular Pemberton method, with the advantages of less danger of molybdic acid depositing and of being able to handle a larger number of determinations at a time. The second difference is in the molybdic solutions, which I consider to the advantage of the official molybdate, and will give my reasons elsewhere. Besides, in adding the official molybdate to the solution to be precipitated, the ammonium molybdate, ammonium nitrate, and nitric acid all go in together, while in the Pemberton procedure they are added separately, with the same result in the end as, I consider, were the formula the same.

ADDITIONAL RESULTS BY VOLUMETRIC METHOD.

Considerable work with the Volumetric method has been done at the New Jersey Experiment Station, and the results reported to me by Mr. L. A. Voorhees follow in Tables VIII and IX. Mr. Voorhees says that, in the work in Table VIII he "used three methods of precipitating the 'yellow precipitate:' first, the one described by Pemberton; second, that described by yourself; and third, by adding sufficient ammonium nitrate (15 grams) and ammonium molybdate (official solution in nitric acid) and allowing to stand, without heating, at the temperature of the room for twelve hours (or over night). This method has been used here in the precipitation for the official method also, and in no case was a second precipitate secured by heating the filtrate with additional ammonium molybdate. Its advantage in the titration method comes in where the system of making strings of analyses is used and one can use his time in removing the supernatent liquid."

TABLE VIII.—*Results by the Volumetric and Official methods on reporter's and other samples.*

	Official.	Pemberton.	Kilgore.	New Jersey station.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
I. Castor pomace and cotton-seed meal	2.62	2.51	2.49	2.45
	2.59	2.51	2.49	2.43
	....	....	....	2.48
	....	....	....	2.45
II. Acid phosphate	17.18	18.15	17.55	17.30
	17.12	18.00	17.33	17.37
III. Solution per 100 grams of P_2O_5	.1999	.2042	20.28	20.19
	.1980	.2054	20.19	20.13
New Jersey samples:				
No. 1. Boneblack superphosphate	15.72	....	....	15.67 15.67
No. 2. Complete fertilizer	8.71	....	....	8.75
No. 3. Complete fertilizer	11.94	....	....	11.87 11.92
No. 4. Complete fertilizer	10.05	....	....	10.00
No. 5. Complete fertilizer	16.96	....	....	16.86 17.12
No. 6. Complete fertilizer	11.59	....	....	11.52
No. 7. Complete fertilizer	9.96	....	....	9.75 9.40
No. 8. Complete fertilizer	10.22	....	....	10.21
No. 9. Complete fertilizer	9.04	....	....	8.90

No. 1, plain boneblack superphosphate.
No. 2, mixture of dissolved bone, KCl, and $NaNO_3$.
No. 3, mixture of $NaNO_3$ $(NH_4)_2SO_4$, blood, fish, bone, boneblack, sup., acid phosphate, and KCl.
No. 4, mixture of $NaNO_3$, tankage, ground bone, boneblack sup., and KCl.
No. 5, mixture of $NaNO_3$ ground bone, boneblack superphosphate, and KCl.
No. 6, mixture of $NaNO_3$ $(NH_4)_2SO_4$, dried blood, boneblack, and KCl.
No. 7, mixture of $NaNO_3$, tankage, dissolved bone, and KCl.
No. 8, mixture of $NaNO_3$, blood, bone, boneblack superphosphate, KCl, and K_2SO_4.
No. 9, mixture of $NaNO_3$, blood, bone, boneblack acid phosphate, KCl and K_2SO_4.

In Table IX are the results of 48 analyses of commercial completes. This work was done by the entire force of the laboratory in the usual rapid way of performing our control work. The results do not agree as well as they should, and I think it is because the titration method requires considerably more care than it received in this instance, at least till it becomes more familiar. The official determinations were duplicated in order to make our reported work satisfactory to ourselves, but there was no time left to duplicate the titrate ones. The results tend to be high by the volumetric method on high percentages especially, but are generally good on low ones.

TABLE IX.—*Results by Pemberton and Official methods on commercial mixed and complete fertilizers.*

	Pemberton.	Official.		Pemberton.	Official.		Pemberton.	Official.
	Per cent.	*Per cent.*		*Per cent.*	*Per cent.*		*Per cent.*	*Per cent.*
10	11.65	11.45	26	9.84	9.56	42	12.02	11.77
11	13.50	13.23	27	7.74	7.68	43	10.31	10.21
12	13.22	13.23	28	9.60	9.30	44	12.80	12.68
13	12.65	12.53	29	12.42	12.12	45	10.24	10.24
14	9.30	9.14	30	12.99	13.10	46	11.95	11.97
15	8.16	7.95	31	13.42	13.06	47	9.23	9.12
16	7.04	7.04	32	11.51	11.15	48	10.44	10.56
17	5.50	5.54	33	11.52	11.16	49	12.12	12.21
18	3.59	3.55	34	11.00	10.88	50	12.93	12.87
19	12.75	12.88	35	10.34	10.03	51	1.62	1.82
20	12.89	12.76	36	15.28	14.84	52	9.30	9.43
21	9.26	9.26	37	12.06	11.99	53	11.62	11.81
22	11.80	11.68	38	7.59	7.62	54	11.25	11.26
23	9.32	9.21	39	9.03	9.08	55	11.58	11.44
24	10.15	9.89	40	8.33	8.21	56	9.18	9.37
25	8.84	8.75	41	10.26	10.26	57	10.83	10.96

WASHING THE MAGNESIUM-AMMONIUM PHOSPHATE.

The results upon this subject on the samples sent out by the reporter, and on others, are brought together in Table X.

TABLE X.—*Washing the magnesium-ammonium phosphate, reporter's samples.*

Analyst.	No. 1.				No. 2.				No. 3.			
	Washed as usual.	Washed free of chlorids (practically).	Times or cubic centimeters as usual.	Times or cubic centimeters to free of chlorids.	Washed as usual.	Washed free of chlorids (practically).	Times or cubic centimeters as usual.	Times or cubic centimeters to free of chlorids.	Washed as usual.	Washed free of chlorids (practically).	Times or cubic centimeters as usual.	Times or cubic centimeters to free of chlorids.
	Per ct.	*Per ct.*			*Per ct.*	*Per ct.*			*Per ct.*	*Per ct.*		
R. E. Noble[1]	2.81	2.55	10	18	18.09	17.81	15	20	20.90	20.50	15	20
	2.75	2.53	10	18	18.15	17.86	15	20	21.08	20.56	15	20
	2.78	2.67	10	18	18.20	17.86	15	20	21.00	20.65	15	20
	2.86	2.64	10	18	18.02	17.88	15	20	......	......	......	......
H. B. McDonnell	2.74	2.58	90	260	17.50	17.42	......	......	20.19	20.03	......	......
	2.72	2.72	......	......	17.47	17.39	120	305	20.29	20.21	105	300
C. L. Hare[1]	2.86	2.58	......	......	18.40	17.90	......	......	......	......	......	......
	2.88	2.61	......	......	18.11	17.88	......	......	......	......	......	......
	2.80	2.67	12	25	18.31	17.90	......	......	......	......	......	......
	2.80	......	......	......	18.43	17.81	12	25	......	......	......	......
C. L. Hare (soluble)[1]	......	......	......	......	12.60	12.13	......	......	......	......	......	......
	......	......	......	......	12.67	12.14	......	......	......	......	......	......
	......	......	......	......	12.63	12.14	......	......	......	......	......	......
	......	......	......	......	12.67	12.14	12	......	......	......	......	......
Means	2.80	2.62	......	......	16.51	16.16	......	......	20.69	20.39	......	......

RESULTS ON OTHER SAMPLES.[2]

Analyst.	No. 1.				No. 2.				No. 3.			
J. S. Meng and R. E. Noble.[1]	14.64	14.16	8	21	......	......	......	......	......	......	......	......
	14.84	14.01										
	......	14.13										
	11.44	10.44	8	21	......	......	......	......	......	......	......	......
		10.48										
		10.45										
	10.14	9.85	10	25	......	......	......	......	......	......	......	......
	10.40	10.00	10	25	......	......	......	......	......	......	......	......

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[1] It is customary with these analysts to wash practically free of chlorids. These experiments were made to the effect of washing.

[2] North Carolina Experiment Station, Technical Bul., No. 7.

COMMENTS OF ANALYSTS ON WASHING THE MAGNESIUM-AMMONIUM PHOSPHATE PRECIPITATE.

R. E. Noble, Alabama.—The results are more uniform and lower when the "white" precipitate is washed free of chlorids than when the washing is only partial.

C. L. Hare.—There is always a considerable diminution in the weight of precipitates washed free of chlorids from those washed only ten or twelve times, and the results are more concordant after freeing of chlorids.

L. A. Voorhees, New Jersey.—The Gooch crucible was used in collecting the "white" precipitate, and after washing, as is our custom, no chlorids were present.

John M. McCandless, Atlanta, Ga.—Filtrations were made in Gooch crucible, and no chlorids were present after washing as usual.

W. A. Powers, Illinois.—It has been my custom to wash the "white" precipitate twenty times, which I have found to free it of chlorids.

F. B. Carpenter, North Carolina.—My experience is that there is more danger of getting high than low results, and that it is necessary to wash free of chlorids.

Reporter.—It is my practice and that of this laboratory to wash all "white" precipitates practically free of chlorids. We have often found differences of from 0.20 to 0.50 per cent between 10 to 12 and 15 to 18 washings on ordinary mixed fertilizers, and much greater on high grade materials. Our method gives high results at best, and they are markedly so unless care is taken in washing.

DISCUSSION OF RESULTS.

The percentages by 10 and 15 washings have been considerably diminished on washing 18 and 20 times, and even the results as shown by No. 3 at least, are much above the true contents of phosphoric acid. The results are also more concordant from having been practically freed of chlorids. It is hardly necessary to say that there is a very great difference in magnesium-ammonium phosphate precipitates, even from the same material and solution, as to the ease or difficulty with which they can be freed of chlorids depending on the excess of magnesia mixture, conditions of precipitation, kind of precipitate, etc. The least magnesia mixture used in order to have an excess the better and the easier the precipitates are to wash clean. I rather suspect that most of us use too much magnesia mixture, which, on long standing in strong alkaline solution, is likely to contaminate our precipitates, giving rise to high results. Of course, where the Gooch crucible is used in filtering little difficulty will be found in freeing of chlorids, and by freeing of chlorids I do not mean the removal of the last trace; this would be tedious and unprofitable, but the precipitate should be practically free of them.

OTHER COMMENTS OF ANALYSTS.

R. E. Noble, Alabama.—For total and insoluble solution in HNO_3 and HCl give equally as good results without as with ammonium nitrate, but for soluble ammonium nitrate is necessary.

H. W. Wiley.—Would it not be well for the association to go back to some of the old methods of getting a white color in the pyrophosphate? Moistening the residue with ammonium nitrate or nitric acid and reigniting once or twice will give a perfectly white material usually.

R. de Roode, West Virginia.—It was found necessary to evaporate the water-soluble of No. 1 (castor pomace and cotton-seed meal) to dryness and ignite in order to get the phosphate in form precipitable by molybdic solution.

When small portions of substance are taken for analysis high results are obtained. Of two portions of the same solution representing ¼ and ½ gram substance, respectively the ½ gram portion will not give twice as much P_2O_5 as the ¼ gram portion. Precipitating 2, 1, and ½ grams of No. 1 gave 2.41, 2.51, and 2.60 per cent, respectively, of P_2O_5. In like manner ½ and ¼ gram No. 2 gave 17.18 and 17.39 per cent of P_2O_5; while on ⅕ and ½ gram No. 3 were obtained 20.07 and 19.91 per cent P_2O_5, respectively, and the results for No. 3 were higher for the small amounts of substances regardless of whether the precipitation was made direct with magnesia mixture or in the usual way with molybdate first. The molybdate method, as carried out by us, in my opinion tends to give high results, and these errors are multiplied when small quantities of substances are operated on and divided where large amounts are used.

B. B. Ross.—Prof. Ross reported, after the results from the Alabama State Laboratory had come in and been tabulated, that he had found the ammonium nitrate used in the work on the reporter's samples to be contaminated with phosphoric acid, but that the salt was not homogeneous and a constant correction could not be gotten. The results from Alabama should be lowered somewhat because of this. He further stated that this was the first time he had found phosphoric acid in ammonium nitrate. To which the reporter would add that once in the laboratory of the North Carolina Station we found our ammonium nitrate considerably contaminated with phosphoric acid.

BURNING THE MAGNESIUM AMMONIUM PHOSPHATE PRECIPITATE.

The official method in this point is to "filter, wash with dilute ammonia, ignite at white heat for ten minutes, and weigh." For small precipitates this may be sufficiently definite, but for large ones I do not think so. I have heard of some chemists who wash and put the wet filter and precipitate into the crucible together and burn. In this way the precipitate forms itself into a mass, burns white on the outside, but how black it is in the interior is not known. The precipitate should be burned white or grayish-white at least.

DILUTE AMMONIA FOR WASHING.

I would also call attention to an error as to equivalence, in the directions for making dilute ammonia for washing. One volume of ammonia, sp. gr. 0.96, mixed with 3 volumes of water, making 4 volumes, is not of the same strength as 1 volume concentrated ammonia, presumably of 0.90 sp. gr. with 6 volumes water; 0.96 sp. gr. ammonia contains practically 10 per cent NH_3 and 0.90 contains 29 per cent. The ammonia wash from 0.96 sp. gr. ammonia would therefore contain 2.5 per cent NH_3, and that from 0.90 sp. gr. 4.14 per cent. Concentrated ammonia of 0.90 sp. gr. with 10 volumes of water contain 2.64 per cent NH_3, being practically of the same strength as that made from the 0.96 sp. gr.

RECOMMENDATIONS AND SUGGESTIONS.

(1) That the directions given in Bulletin 38, Chemical Division, U. S. Department of Agriculture, page 170, second to last line in last paragraph, under total phosphoric acid for washing and burning the magnesium ammonium phosphate precipitate, be made to read: "Filter, wash with dilute ammonia until practically free of chlorids, ignite till precipitate is white or grayish white, and weigh."

(2) That the directions for making dilute ammonia wash be made to read: "One volume of ammonia, sp. gr. 0.96, mixed with 3 volumes of water, or 1 volume 0.90 sp. gr. ammonia with 10 volumes of water."

(3) In the directions for making the solution of the citrate-insoluble, after the word "dissolved," in line 14, under the head of "Citrate-insoluble phosphoric acid" (p. 169, Bull. 38, U. S. Dept. Agr.), add, "or transfer the filter and insoluble residue back to the digestion flask or to other suitable flask," add 30 to 35 cc. nitric acid and 5 to 10 cc. hydrochloric acid, and boil for forty-five to sixty minutes.

(4) While the Volumetric method in its different forms has given encouraging results and almost universally good ones on low percentages, I do not consider that anything should be done with it beyond making it the subject of further investigation.

(5) I suggest that the addition of a small amount of citric acid to the alkaline solution from which the "white precipitate" is formed will probably add to the accuracy of the results, but am not sure that this will be practicable on account of the fineness of the precipitate and its liability to run through the filter.

At the conclusion of Mr. Kilgore's report the presiding officer called for any papers which were to be presented on the subject of phosphoric acid.

Mr. Ross stated that he had a paper on a new reaction of phosphomolybdate of ammonia.

In connection with the paper which follows, Mr. Ross presented samples showing the character of the reaction.

A NEW REACTION WITH AMMONIUM PHOSPHOMOLYBDATE.

By B. B. Ross.

Baumann (Ztschr. f. anal. Chem., 1892) and others have called attention to the reaction which takes place when chromates are brought in contact with dilute acids and hydrogen dioxid, and have proposed a number of analytical methods based upon this reaction.

On account of the analogy which is known to exist, at least to a limited extent, between chromates and molybdates, it was determined to test the influence of reagents of this character upon some of the salts of molybdic acid.

An aqueous solution of ammonium molybdate, when treated with dilute sulphuric acid and hydrogen dioxid and heated, almost immediately gave a bright yellow solution, which failed to precipitate a trace of phosphoric acid from a solution of a chemically pure phosphate. A sample of freshly precipitated ammonium phosphomolybdate was next tested with the same reagents and it was found that its decomposition took place quite slowly in the cold. Under the influence of heat, however, the solution of the phosphomolybdate took place rapidly and completely, a bright yellow solution being secured as before. The best results were secured by placing the well-washed yellow precipitate, together with the filter, in the flask or beaker used in the precipitation, and adding about 50 or 60 cc. boiling dilute sulphuric acid and about 30 or 40 cc. hydrogen dioxid; heat is then applied and the liquid is boiled until a clear solution is secured, about three minutes being generally required for this purpose. Sufficient time has not been at hand in which to determine with certainty the nature of the reaction which takes place, but the phosphoric acid is probably present as ammonium phosphate, while the molybdic acid has apparently been converted into one of the molybdenum sulphates.

It is believed that this reaction may possibly prove of some value as a basis for the volumetric determination of phosphoric acid.

A number of experiments with this end in view is now in progress and more definite results, it is believed, would already have been secured had it not been that great difficulty has been experienced in obtaining hydrogen dioxid of a sufficient degree of purity.

The presiding officer called for further papers.

Mr. Kilgore stated that he had one or two papers on some outside work which he had done in the Volumetric method and which he thought would be of interest in connection with the very general interest which is taken at this time in volumetric work.

Mr. Kilgore then read a paper by Francis Bergami, of Philadelphia.

COMPARISON OF PEMBERTON'S METHOD OF PHOSPHORIC ACID DETERMINATION WITH THE OFFICIAL METHOD BY FRANCIS BERGAMI.

By the kindness of Dr. Terne I came into the possession of a copy of Mr. Pemberton's paper on phosphoric acid determination. The results shown therein indicate such a surprising accuracy of the method that Dr. Terne requested me to start an investigation at once, as it would be of the highest importance for our work to find a more rapid but, at the same time, equally accurate process of phosphoric acid determination than the Official method. As my spare time is very limited I have extended my tests only to such materials as just at present enter our laboratory for examination. These materials consist mostly of ammoniated and mixed phosphates.

The results of all the tests made up to this time are given in the following table:

Phosphoric acid.

Sample.	Official method.	Pemberton method.	Difference.
	Per cent.	*Per cent.*	*Per ct.*
1. Ammoniated phosphate	11.84	11.68	0.16
	....	11.77	.07
2. Ammoniated phosphate	13.20	13.05	.15
	....	13.14	.06
3. Ammoniated phosphate	8.80	8.68	.12
4. Ammoniated phosphate	8.74	8.74	....
5. Dissolved boneblack	20.68	20.56	.12
	....	10.93	.03
6. Ammoniated phosphate: water-soluble phosphoric acid	10.96	10.84	.12
7. Ammoniated phosphate	12.48	12.35	.13
8. Dissolved boneblack not identical with No. 5	20.68	20.55	.13
9. Acidulated South Carolina rock:			
Total phosphoric acid	15.52	15.34	.18
Insoluble phosphoric acid	1.72	1.68	.04
Available phosphoric acid	13,80	13.66	.14
10. Ammoniated phosphate	12.32	12.17	.15
11. Ammoniated phosphate: water-soluble phosphoric acid	10.56	10.47	.09
12. Acidulated South Carolina rock:			
Total phosphoric acid	15 29	15.16	.13
Insoluble phosphoric acid	2.56	2.49	.07
Available phosphoric acid	12.73	12.67	.06
13. Refuse boneblack	34.40	34.03	.37
	....	33.94	.46
	....	34.03	.37
14. South Carolina rock	27.66	27.34	.32
15. Florida rock	32.24	31.94	.30

In reviewing all the results it can be seen that, on the average, the Pemberton process gives a little lower results than the Official method. Supposing my work is as free from individual error as any man's work can be, there are only two explanations for the differences; either the results of the Official method are a little too high or those of the Pemberton method a little too low. I feel very much inclined to accept the first explanation, for the following reason:

It has been repeatedly stated by different chemists that it is hardly possible to obtain by one precipitation a precipitate of ammonium magnesium phosphate absolutely free from magnesium oxid, and it has been recommended to dissolve the precipitate again and reprecipitate it by ammonia.

Dr. N. von Lorenz has only lately published his investigation in regard to this source of error in the Official method (FRESENIUS Zeitschrift, 1893, p. 64). He shows that the addition of about 2 per cent of citric acid to the solution of the phosphomolybdate in ammonia will prevent any contamination by magnesium oxid.

When I had obtained the results on sample No. 13, a refuse boneblack, I found the difference between the official test and even the highest result of the Pemberton method, namely 0.37 per cent, too high to be passed without closer consideration. In this case I had accidentally omitted the neutralization of the ammoniacal solution of the phospho-molybdate by hydrochloric acid. Therefore the idea struck me that this neglect might be the cause of too high a result and I concluded to repeat the test twice with addition of citric acid, as recommended by Dr. von Lorenz. The results thus obtained were 34.08 and 34.16 per cent against 34.03 and 33.94 per cent by Pemberton's method and 34.40 per cent by Official method without neutralization. In former years I had always precipitated without previous neutralization of the ammonia solution, but as I have lately followed strictly the Official way and neutralized, I thought surely that the omission of neutralization was the cause of the high result. The smaller differences in all previous tests could easily be explained as individual working errors, but when I obtained the results in samples Nos. 14 and 15, I felt a little astonished.

In these cases I was sure of no neglect on my side; the official way was carefully carried out as in all other samples and I had made the solutions of the yellow precipitate as nearly neutral as possible. Still the differences between both methods were 0.30 and 0.32 per cent, respectively.

I always wash the ammonium magnesium phosphate until the filtrate shows no chlorin reaction, so that the source of error arising from incomplete washing is absolutely excluded and I am therefore at a loss to find any other explanation for the differences than that even in nearly neutralized solutions a small amount of magnesium oxid is thrown down together with the ammonium magnesium phosphate and renders the results of the official method too high. Naturally the error will show itself more in high-grade phosphates than in those with lower percentages.

In calculating the results I used the figures obtained by Mr. Pemberton, which are based upon the fact that 23 molecules of alkali are necessary to neutralize 1 molecule of ammonium phospho-molybdate. In establishing this fact Mr. Pemberton precipitates a solution of disodic hydric phosphate by magnesium mixture, but he obtains a precipitate absolutely free from magnesium oxid by the precaution to dissolve the first precipitate again and reprecipitate with ammonia. In this way he eliminates the above-stated error in the Official method.

If in establishing the relation between the phosphoric acid in the yellow precipitate and the standard alkali solution the ammonium magnesium phosphate would be weighed right after the first precipitation, I am nearly sure the figures thus obtained would be higher and results in analyses calculated on those figures would not show such big differences from the Official method, as they would involve the same source of error.

In regard to the execution of the Pemberton method, I should like to state the following:

Extremely great care must be taken in washing the yellow precipitate until free from acid. Testing with delicate litmus paper should never be omitted. In regard to rapidity I found it of some advantage to introduce a little change in Mr. Pemberton's way.

To the 25 cc. of acid solution, which is always obtained in the same way (1 gram substance, 35 cc. nitric acid and 5 cc. of hydrochloric acid), I add at once 10 cc. of strong ammonia, sp. gr. 0.90, and after a little stirring 15 cc. of nitric acid, sp. gr. 1.42.

This operation secures about the same conditions for all tests and produces at the same time a temperature of 75° to 80° Celsius, which reduces the time required for heating to boiling point to a minimum.

The method is absolutely free from all difficult manipulations and the only inconvenience I experienced with it occurred in such samples as gave dark-colored acid solutions. A considerable part of the coloring matter enters the precipitate and imparts to the solution in alkali a brownish color. This renders the titration a little difficult and may in some cases give rise to uncertainty and error. A previous ignition of the sample or treating with concentrated sulphuric acid would overcome this difficulty. Whether in the case of solution in sulphuric acid a larger amount of nitrate of ammonia and a longer allowance of time for complete precipitation would be necessary is still an open question and needs investigation.

Mr. Pemberton recommends a standard alkali solution which is free from carbonic acid. I have found that in the determination of nitrogen and ammonia a solution with a small amount of carbonates works just as well as if free from the same, provided phenolphthalein and cochineal are used as indicators for standardization and analysis, respectively. Both indicators show absolutely sharp end reactions, even if a small amount of carbonates is present. Curious of seeing if carbonic acid would have any detrimental effect in this special case, I repeated some of the tests with an old soda solution containing some carbonates with the following results:

	Sample.				
	1.	4.	5.	12.	15.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Alkali with CO_2	11.68	8.69	20.47	15.07	31.94
Alkali without CO_2	11.77 11.68	8.74	20.56	15.16	31.94 ....

The close agreement of the different results seems to show that the precipitation of the carbonic acid is an unnecessary precaution.

I do not consider the number of tests made sufficient to prove the absolute infallibility of the Pemberton method, but I think that my work shows at least the probability of obtaining fair results by the same.

The results obtained by Mr. Pemberton and myself should be encouraging enough for any chemist employed in the control of fertilizers to make a careful study of the method with the hope of producing an absolute proof of accuracy which would justify the abandonment of the old, tedious, and time-wasting way of the Official method.

ON THE ESTIMATION OF PHOSPHORIC ACID BY TITRATION OF THE YELLOW PRECIPITATE OF PHOSPHOMOLYBDATE OF AMMONIA.

By B. W. Kilgore.

Methods based upon the principle of titrating the yellow precipitate with standard alkali have been described by a number of chemists, especially for the estimation of phosphorus in iron and steel and their ores, where it is present in very small quantities.

Among them may be mentioned, by abstract, E. Thilo (abs. Jour. An. Chem., Vol. I, part 2, p. 219, 1887), in the estimation of P_2O_5 in Thomas slag, precipitates at 70° C. with molybdic solution containing 1 part molybdic acid, 3 parts ammonium nitrate, and 15 parts nitric acid, allows to stand five minutes, filters, washes with 20 per cent ammonium nitrate solution, and titrates with ammonium hydrate and back with sulphuric acid.

C. E. Manly (Jour. An. and App. Chem., 6, 82) precipitates near boiling, washes with 3 per cent each nitric acid and potassium nitrate, dissolves in 15 per cent ammonium hydrate, boils off the excess of ammonia, adds 2 cc. of nitric acid of 1.20 sp. gr., evaporates to dryness, heats sufficiently to expel nitric acid and ammonium nitrate, titrates with sodium hydrate and back with nitric acid.

J. O. Handy (Jour. An. and App. Chem., 6, 204) precipitates at 85° C., shakes for five minutes, filters, washes five times with 1 per cent nitric acid, five times with 0.1 per cent potassium nitrate, and titrates.

M. Rothberg and W. A. Auchinvole (Jour. An. and App. Chem., 6, 243) precipitate at 40° C., shake for one minute, allow to stand in a warm place five more, filter, wash five times with 1 per cent nitric acid, five times with 0.3 per cent potassium nitrate, titrate with sodium hydrate, and back with nitric acid.

Dr. Franz Hundeshagen (Chem. News, Vol. 60, pp. 168, 177, 188, 201, 215) in his analytical studies of phospho-molybdate of ammonia shows its composition under all conditions, when free of molybdic acid and other impurities, to be $12MoO_3PO_43NH_4$. He washes the precipitate with a 5 per cent solution of ammonium nitrate, titrates with sodium hydrate and back with nitric acid. He found that it required 23 molecules of alkali to neutralize 1 molecule of phospho-molybdate of ammonia.

H. Pemberton, jr. (Jour. Am. Chem. Soc., Vol. xv, No. 7, p. 382), has published the description of a method on this principle which he has applied to the determination of phosphoric acid in high-grade phosphates. He uses 0.1 gram of substance for analysis, precipitates at boiling with aqueous ammonium molybdate, washes with water, titrates with potassium hydrate free from carbonates and back with hydrochloric acid. In his first work he found that it required 23.2 molecules of alkali to neutralize 1 molecule of ammonium phospho-molybdate, but in his later work he has obtained 23 molecules.

The writer has made a number of determinations of phosphoric acid in materials of varying percentages by this method with fairly good results generally, but with wide variations at times, the tendency of the method being to give high results. The yellow precipitate was washed liberally to remove free acid. It was believed that molybdic acid was the cause of the variations, as it was known that molybdic acid would deposit from other molybdic solutions very quickly at the temperature of precipitation used in the Pemberton method. To see if the aqueous molybdic was an exception to this rule, and also to see if the official molybdate would take the flow of the aqueous solution, the official molybdate was used for precipitating at boiling temperature, the method as described by Pemberton being carried out otherwise, except that 0.2 gram of substance was used. Results obtained in this way were less subject to variations than when the aqueous molybdate was employed, though high percentages would still be gotten in many cases. Both molybdates were then tried side by side in the water bath at temperatures of 70°, 65°, and 60° C. (this means, practically, 5° lower temperature in the beaker), allowing five minutes for precipitation, which was found in all cases to be sufficient. The results thus obtained were favorable to the lower temperature of 60° C. in bath and to the official molybdate, there being very few variations from the Gravimetric method when precipitation was made at 60° C. and with Official molybdate.

A great many results were obtained in testing these and other points, but most of the early ones were not kept. In the following table are brought together some of the earlier results and all of the later ones by the Volumetric method, either as carried out by Pemberton or myself or both, in the order in which they were obtained, and also results by the Gravimetric method on the same samples for comparison. The Gravimetric results were not duplicated. Among the earlier results by the Volumetric method there are quite a number of wide variations from the Gravimetric results, but the later ones by the Volumetric method, as carried out by myself, nearly all show close agreement with Gravimetric results.

TABLE I.—*Determinations of phosphoric acid in various fertilizers by the Gravimetric method and by the Volumetric method as carried out by Pemberton and as carried out by Kilgore.*

No.	Official.	Pemberton.	Kilgore.	No.	Official.	Pemberton.	Kilgore.
	Per cent.	*Per cent.*	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*
1	10.40		[1]10.34	44	9.12		[2]9.17
2	9.48		[1]9.40	45	10.20		[2]10.33
2			[1]10.52	46	13.70		[2]13.62
3	2.44		[1]2.53	47	13.92		[2]13.92
4	2.85		[1]2.78	48	15.75		[2]15.50
5	3.90		[1]3.83	49	8.14		[2]8.40
5			[1]3.80	50	8.40		[2]8.22
6	.08		[1].30	51	10.73		[2]10.80
7	2.17	2.18		52	9.63		[2]9.67
8	2.72		[1]2.62	53	10.50		[2]10.48
9	3.76	3.76		54	10.53		[2]10.58
10	15.58		[1]15.48	55	11.13		[4]11.10
10			[1]15.40	56	8.96		[4]8.87
11	14.09		[2]13.96	57	10.75		[4]10.72
12	13.61		[2]13.36	58	10.73		[4]10.74
12			[2]13.60	59	11.63		[4]11.53
13	12.98		[1]13.06	60	9.59		[3]9.64
14	9.79	10.60	[2]9.63	61	13.96		[3]14.18
14			[2]9.70	62	14.43		[6]14.33
15	9.95	10.25	[2]9.90	63	14.37		[6]14.23
15		10.20	[2]9.83	64	10.67		[3]10.65
16	1.25		[2]1.14	65	9.49		[3]9.55
17	2.64		[2]2.56	66	10.61		[3]10.77
18	1.68		[2]1.58	67	10.93		[3]11.11
19	4.06	4.05		68	11.55		[6]11.59
20	2.07	2.10		69	12.60		[6]12.45
21	1.37	[2]1.31		70	10.16		[6]10.45
22	2.14	[2]2.19		71	14.80		[6]14.77
23	1.30		[2]1.23	72	9.95		[2]10.20
24	3.93		[2]3.93	73	9.93		[2]10.22
25	10.90	10.87		74	9.63		[2]9.80
26	11.28	[2]11.90		75	10.52		[7]10.49
27	8.89	10.55	[2]9.00	76	11.00		[2]10.98
28	10.61	10.98		77	10.68		[7]10.71
29	9.93	10.14		78	13.42		[7]13.26
30	9.72	9.98		79	12.63		[7]12.60
31	10.59	10.96		80	15.06		[7]14.89
32	9.04		[3]9.10	81	14.79		[7]14.84
33	8.72		[3]8.72	82	14.87		[7]14.84
34	10.07		[4]10.00	83	12.22		[7]12.17
35	10.41		[4]10.26	84	12.75		[2]12.72
36	10.43		[5]9.46	85	12.46		[7]12.34
37	12.26		[6]9.94	86	11.23		[7]11.09
38	8.83		[3]8.84	87	9.03		[7]9.12
39	8.11		[4]8.08	88	10.49		[7]10.40
40	13.26		[5]8.87	89	15.43		[7]15.30
41	10.59		[3]10.34	90	16.27		[7]15.86
42	8.83		[4]8.56	91	15.98		[7]15.61
43	9.17		[3]9.26	92	15.71		[7]15.71

[1] Precipitated at boiling.

[2] Precipitated in bath at 70° C.

[3] Precipitated in bath at 65° C.

[4] Precipitated in bath at 65° C., with the addition of 5 cc. HNO_3 to 50 cc. molybdic solution (official).

[5] Same as (4) with 10 cc. HNO_3.

[6] Same as (4) with 3 cc. HNO_3.

[7] Precipitated in bath at 60° C.

TABLE I.—*Determinations of phosphoric acid in various fertilizers, etc.*—Continued.

No.	Official.	Pemberton.	Kilgore.	No.	Official.	Pemberton.	Kilgore.
	Per cent.	*Per cent.*	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*
93	11.83	....	[1]11.63	107	15.20	16.17	[2]15.25
94	10.04	....	[1]9.94	108	13.96	....	[1]14.18
95	13.28	....	[1]13.25	109	13.60	....	[1]13.67
96	10.55	....	[1]10.42	110	15.67	....	[1]15.82
97	10.41	....	[1]10.40	111	11.80	....	[1]11.85
98	9.76	....	[1]9.62	112	11.74	....	[1]11.75
99	11.26	....	[2]11.25	113[3]	19.93	20.35	[1]19.85
100	11.49	....	[1]11.37		19.83	20.15	[1]19.95
101	15.83	....	[1]15.73		19.91	20.15	[1]19.90
102	15.71	....	[1]15.71		19.85	20.10	[1]19.80
103	15.25	....	[1]15.20	114	17.17	17.20	[1]17.15
104	11.64	12.15	[2]11.63		17.12	17.40	[1]17.10
105	15.89	16.20	[2]15.71	115	2.42	....	[1]2.47
106	15.54	16.15	[1]15.65				

[1] Precipitated in bath at 60° C.

[2] Precipitated in bath at 70° C.

[3] C. p. $Na_2HPO_4 + 12\ H_2O = 19.826$ per cent P_2O_5.

There are two main differences in the Volumetric method as carried out by Mr. Pemberton and as carried out by myself. First, the temperature of precipitation in the former is at boiling; in the latter 60° C. in the bath or 55° C. in the beaker. At the former temperature molybdic acid deposits very quickly, especially if any considerable excess of the precipitant is present. If we know how much phosphoric acid our sample contains and add just enough molybdate for complete precipitation, or are willing to add little by little of the precipitating reagent till the proper amount has been added, this objection is largely overcome, but the former we do not know and the latter is tedious and time-consuming, especially where large numbers of analyses are being carried on at once.

At 55° C. the official molybdate does not deposit for fifteen minutes or more; at worst, precipitations always complete in four or five minutes from the time the molybdate is added, and precipitation and filtration can be finished in almost if not in as short a time by this method of procedure as by the regular Pemberton method, with the advantages of less danger of molybdic acid depositing and of being able to handle a larger number of determinations at once.

The second difference is in the two molybdic solutions. The official molybdic solution contains practically 60 grams MoO_3 to the liter, and the Pemberton one 66 grams to the liter, so there is no very great difference in the precipitating power. In the official molybdate the proportion of free nitric acid to molybdic acid is always the same, being equivalent to nearly 15 cc. 1.42 sp. gr. acid to 50 cc. molybdic solution; while in Pemberton's molybdate the quantity is just 5 cc., 1.42 sp. gr., nitric acid, no matter whether 25 or 50 cc. of the aqueous molybdate is added. It will be readily understood that should any considerable excess of this aqueous molybdate be added (and such a thing is liable to be done), there being only 5 cc. nitric acid to hold it in solution, precipitation would be sure and quick.

To see which of the two molybdates was more liable to deposit molybdic acid under the same conditions, the conditions being as nearly as possible those of precipitation without the phosphate, the following experiments were made: Fifty cubic centimeters of official molybdate, containing 3 grams MoO_3 and 15 cc. HNO_3 were diluted with 50 cc. of water and 45 cc. of Pemberton's molybdate, containing 2.97 grams MoO_3, 10 cc. saturated solution of ammonium nitrate, and 5 cc. nitric acid were diluted with 40 cc. water. Both mixtures, being of the same volume and containing the same amount of molybdic acid, were placed in a water bath at 60° C. The mixture of official molybdate showed no deposit for over twenty minutes, but a considerable one in one hour; the other mixture deposited seemingly the whole content of molybdic acid in less than four minutes, and in fact this mixture could not be made to stand in the cold over three or four minutes without throwing down great volumes of molybdic acid. But 45 and 50 cc. of the two molybdates are too much to precipitate the phosphoric acid from 0.2-gram substance, but not much in excess of safe quantities for most materials where 0.4-gram substance is used.

With 0.2-gram substance one-half the above quantities is about the right amount to use, and experiments with them similar to the foregoing were made as follows: 25 cc. official molybdate containing 1.5 grams MoO_3 and 7.5 cc. HNO_3 were diluted with 25 cc. water; and 22.5 cc. of Pemberton's molybdate, containing 1.48 grams MoO_3, 5 cc. HNO_3 and 10 cc. ammonium nitrate solution, were diluted with 12.5 cc. water. Both mixtures, being of the same volume and containing the same amount of molybdic acid, were allowed to stand as before. The mixture of official molybdate deposited about as in the first experiment; the Pemberton solution remained clear for 10 or 15 minutes, when it commenced to get cloudy, and in 20 minutes it was milky and a heavy deposit had formed. Seventeen cc. of the official molybdate and 15 cc. of the Pemberton solution contain the same amounts of MoO_3, and when these quantities are used for precipitating equal amounts of nitric acid, 5 cc. are present in each case. When these amounts were diluted to 50 cc. and allowed to stand at 60° C. the deposit was the same as in the official molybdate in the two preceding experiments, which argues that the official molybdate contains about the right amount of nitric acid to prevent deposit of molybdic acid, and at the same time to allow quick precipitation of phosphoric acid.

The aqueous molybdate as used in the Pemberton method works satisfactorily when added in just the proper quantity, but there is not enough free nitric acid present to allow of much marginal excess. The official molybdate seems to contain a very large amount of free nitric acid. To test the effect of still larger quantities of free acid, the results in the table, as designated by the footnotes, show that 10 cc. additional nitric acid of 1.42 sp. gr. to 50 cc. of molybdate hindered precipitation greatly, 5 cc. prevented its being complete in five minutes, while 3 cc. additional did not seem to interfere. The more free nitric acid present, not to interfere with precipitation, the less liability there is of molybdic acid depositing.

Just here I would add that the deposit from molybdic solutions is not molybdic acid alone, but a mixture of molybdic acid and ammonium molybdate. I found one deposit to contain 1.21 per cent NH_3.

The Volumetric method, as it has given me best results, is as follows: Make solution as per the official nitric and hydrochloric acids method for total phosphoric acid, measure out 20 cc. for total or 40 cc. for insoluble phosphoric acid, corresponding to 0.2 and 0.4 gram, respectively, into a 4-ounce beaker, add ammonia till a precipitate just begins to form, and dilute to 75 cc. If much of the nitric acid was driven off in making the solution, add 10 to 15 grams ammonium nitrate, otherwise this is not necessary. Digest in water bath at 60° C., and after filtering the molybdate used in the Official method, precipitate in the usual way; allow to stand four or five minutes from the time the molybdate is added, filter as quickly as possible upon either a filter made by putting a platinum cone or disk well filled with holes into a 3 inch funnel and covering with coarse asbestos, or upon the Hirsch funnel, or preferably upon a porcelain disk (the disk to be covered with filter paper) with rubber rim in 3-inch glass funnel, using the pump in all cases; wash three or four times by decantation, using 50 to 75 cc. of water each time and agitating thoroughly; then on the filter until no longer acid, titrate with potassium hydrate and back with nitric acid.

DETERMINATION OF PHOSPHORIC ACID BY THE MOLYBDATE METHOD.

By W. E. Garrigues.

If any apology is necessary for adding to the already voluminous literature on the determination of phosphoric acid, the wide discrepancies in the findings of the different association chemists on four samples sent out by the reporter in 1892, should be ample. The extremes being in per cent P_2O_5 1.45, 1.80, 1.28, 1.46, respectively. From my own experience I can cite a recent difference of over 2 per cent among five commercial chemists of undoubted repute. These specific instances seem to stand out as urgent advocates of an investigation of the accepted methods of analysis.

The work here submitted to the association bears on the three steps in the molybdate method, i. e., the solution, the yellow precipitate, the magnesia precipitate. No claim is put forth that the conclusions arrived at in the interpretation of the results indicate a method which gives an acurate determination of the phosphoric acid present; such was not the object in undertaking the work; it does, however, appear that the points mentioned are prevalent factors in the variation of results by different chemists, presumably working by the same method.

Although the second step in the analysis, that of obtaining the phosphoric acid as ammonium phosphomolybdate is probably responsible for most of the trouble, we will begin with the solution and consider the others in the order in which they occur. The directions given in the official bulletin do not include evaporation to dryness of the phosphate solution to render silica insoluble, but as so many textbooks lay special stress on the importance of so doing, it is likely that this is much practiced and it may not be out of place to mention it here.

To definitely prove that the reactions $H_3PO_4-H_2O=HPO_3$, and $2H_3PO_4-H_2O=H_4P_2O_7$ are not only theoretically correct, but actually do take place when the acid solution of calcium phosphate is evaporated to dryness at temperatures above 100° C., and that the meta and pyro phosphoric acid formed is subsequently only incompletely reconverted to ortho, thereby escaping precipitation with the molybdic reagent, a number of portions of such a solution containing no silica were pipetted off and evaporated with the following result:

On plate at 145°C., per cent P_2O_5.	On water bath, per cent P_2O_5.	Without evaporation, per cent P_2O_5.
24.28	25.38	25.58
24.30	25.57	25.52
24.30	25.58	25.57
24.97	....	25.44
24.34	....	25.39
24.80	....	25.58
Average: 24.49	25.51	25.51

Naturally this shortage will vary between wide limits, according to the temperature at which evaporation is conducted, the length of time boiling is continued to redissolve, and the amount of free nitric acid present with the molybdic solution. The figures are only important as showing what can readily take place under conditions not unusual.

We will now consider only the manipulation adopted by the association as published in Bulletin No. 38. On page 170 we find the following: To the hot solutions for every decigram of P_2O_5 that is present add 50 cc. molybdic solution. The results given below, and taken as typical from many others, seem to warrant the conclusion that while the proportion of 50 cc. molybdic solution to 0.1 gram P_2O_5 is ample for quantities less than that, a much greater excess must be used in case of larger quantities to insure complete precipitation. For instance, 40 cc. of molybdic solution will in one hour completely precipitate the P_2O_5 in 0.25 gram rich Florida phosphate, but 80 cc. will not precipitate it in 0.5 gram even in twice that time.

Aqua regia solutions of a Carolina and two Florida rocks were made and all the portions withdrawn by accurate pipettes; varying quantities of molybdic solution were added in the usual manner and the precipitate allowed to settle in a steam closet at just 65° C. Those given in columns 1 and 4 stood one hour before filtering; those in 2 and 3, two hours.

	1. 0.25 gram, 35 cc. molybdate.	2. 0.25 gram, 70 cc. molybdate.	3. 0.5 gram, 70 cc. molybdate.	4. 0.5 gram, 140 cc. molybdate.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
	26.32	26.22	25.80	26.64
	26.58	26.22	25.79	26.13
	26.22	26.55	25.89	26.32
	26.58	26.58	25.84	26.21
	26.58	26.40	25.44	26.53
	26.41	26.57	25.65	26.19
Average:	26.45	26.42	25.73	26.33
	40 cc.	80 cc.	80 cc.	160 cc.
	30.57	30.56	29.98	30.06
	30.72	30.63	29.80	30.30
	30.56	....	29.75	30.08
	30.73	....	30.00	30.02
	30.73	....	29.47	30.40
	30.65	....	29.45	30.28
Average:	30.66	30.59	29.74	30.19
	28.50	28.51	27.99	28.16
	28.47	28.40	27.65	28.21
	28.32	....	27.46	28.32
	28.45	....	27.93	28.15
Average:	28.43	28.45	27.76	28.21

Here we have an average loss of 0.72, 0.92, 0.67, respectively, in column 3, due entirely to an incomplete precipitation of ammonium phosphomolybdate, although the proportion of 50 cc. of the reagent to 1 decigram of P_2O_5 was strictly adhered to. Column 2 would indicate that a sufficiency had been used in column 1. Four, though some of the results compare favorably with number 1, is lower on an average, and certainly argues against the use of large quantities of the sample, at least for one hour's settling of the yellow precipitate.

In the Journal of the American Chemical Society, July, 1893, Mr. Pemberton gives a volumetric method for the analysis of phosphates, wherein he titrates the yellow precipitate with standard alkali. He uses a neutral solution of ammonium molybdate, 90 grams to the liter, each cubic centimeter being sufficient to throw down 0.003 gram P_2O_5. The reagent is added to the phosphate solution at boiling temperature and the precipitate is filtered out at once.

This plan has been tried in connection with the Official gravimetric method, and has yielded results that, for accordance and rapidity, leave nothing to be desired. Besides the saving of time in the analysis the reagent is so much easier to prepare than the molybdic solution. The fact, however, that large quantities are not completely precipitated holds good again, as with the official reagent, although the discrepancy is not so large. The figures obtained on the same three samples are compared to those given before in column 1.

0.25 gram, 35 cc. official molybdic sol.	0.25 gram, 25 cc. Pemberton's sol.	0.5 gram, 50 cc. Pemberton's sol.
Per cent.	*Per cent.*	*Per cent.*
26.32	26.58	26.05
26.58	26.58	26.13
26.22	26.58	26.03
26.58	26.52	26.30
26.58	26.60	26.01
26.41	26.60	26.22
Average: 26.45	26.57	26.12
40 cc.	27 cc.	54 cc.
30.57	30.73	30.30
30.72	30.72	30.33
30.56	30.69	30.52
30.73	30.80	30.40
30.73	30.70	30.27
30.65	30.70	30.15
Average: 30.66	30.72	30.33
28.50	28.48	28.00
28.47	28.48	28.13
28.32	28.60	27.92
28.45	28 45	27.98
Average: 28.43	28.50	28.01

These results average a little higher and are likewise more accordant than those obtained by the official reagent. This is no doubt due to an occasional incomplete precipitation by the latter, which is apparently unavoidable if the solution is filtered in one hour. All the foregoing precipitates were washed with 2 per cent nitric acid. Water was tried, but with the larger precipitates a little iron frequently remained in the filter after dissolving the ammonium phosphomolybdate. How much of an error this involved was not determined.

Passing on to the final precipitation, the association method gives: Dissolve the precipitate on the filter with ammonia and hot water, nearly neutralize with hydrochloric acid, cool, add magnesia mixture, etc.

No indication is given of the degree of alkalinity that should remain. As a matter of fact, the condition will by most chemists be judged from the difficulty with which the precipitate thrown down by the acid redissolves. It is thus easily possible to get an acid solution or one so nearly neutral that the ammonium magnesium phosphate will not precipitate until more ammonia is added later. In consequence of this there results a flocculent basic precipitate, as was proved by dissolving the filtered and washed ammonium magnesium phosphate in hydrochloric acid and reprecipitating with ammonia, after the addition of a little hydrogen disodium phosphate. There was invariably a further increase in weight by this operation.

On the other hand, whether a considerable or only a slight excess of ammonia is present at the time of adding the magnesia mixture does not appear to have any bearing on the result. The figures in column 1 were obtained by adding magnesia mixture to a solution which was in such a condition that no precipitation took place until more ammonia was added later. In column 2 the solution contained about 1 cc. excess of strong ammonium hydrate. The precipitation then took place slowly while the whole was being stirred. In column 3 there was present an excess of 20 cc. strong ammonium hydrate, and the precipitation began on the addition of the first drop of magnesia mixture.

	1.	2.	3.
	Per cent.	*Per cent.*	*Per cent.*
	26.06	25.57	25.50
	26.23	25.58	25.53
	26.40	25.44	25.79
	26.13	25.39	25.71
	26.05	25.52	25.42
Average:	26.17	25.50	25.59

In conclusion, I can do no better than to state in detail the method which gave at least the most satisfactory results, and which it is believed will, if followed, eliminate much of the chance of differences between chemists engaged in this work.

The phosphate solution, containing not over 0.075 gram P_2O_5, is made just alkaline with ammonia and cleared with 10 cc. nitric acid, sp. gr. 1.42; 20 cc. saturated solution of ammonium nitrate are added, and the whole brought to a boil. Remove from flame and run in at once, with stirring, 25 cc. neutral solution of ammonium molybdate—90 grams in 1 liter. As soon as the precipitate settles, filter and wash with 2 per cent nitric acid; dissolve through the paper in ammonia and hot water. Add hydrochloric acid until a permanent precipitate appears, avoiding an excess; then 20 cc. ammonium hydrate, sp. gr. 0.90. Cool, add magnesia mixture, drop by drop, with constant stirring. (No further ammonia need be added.) When precipitation is complete—several hours—filter, wash with dilute ammonia, transfer while wet to a deep platinum crucible and ignite gently until the paper is charred. Raise the flame to the full red heat of a Bunsen burner, remove the crucible cover, and allow the paper to burn completely away. Replace the lid, leaving a small opening on one side, and finish the ignition with a blast burner, attaining a white heat. The $Mg_2P_2O_7$ should be a spotless white and of constant weight.

COMPARISON OF RESULTS BY OLD AND NEW METHODS FOR CITRATE-INSOLUBLE PHOSPHORIC ACID.

By B. W. Kilgore and C. B. Williams.

The change in the temperature of digestion and otherwise in the manipulation of the old method of 1893 and back for citrate-insoluble phosphoric acid, caused by the adoption of the modification of the method at the last meeting of this association, has carried with it considerable lowering of the results for citrate-insoluble. The increase in temperature amounts to about 2° C.; for a bath that formerly stood at 65° C. must now be raised to 67° or 68° to give 68° in open flask, and 65° in open flask corresponds to about 66° in the closed one, so that the digestion is really made at 66° C.; and it is to this increase of temperature and time of digestion we consider that the lowering of the results is due.

In the accompanying table are presented comparisons of some results by the old method and as modified, obtained in the laboratory of the North Carolina Experiment Station previous to changing to the new method. The differences are considerable, but do not represent the full magnitude of some we have obtained and heard of, which have amounted to more than 1 per cent.

Results by old and new methods for citrate-insoluble phosphoric acid.

Nos.	New method, 1894.	Old method, 1893.	Difference less by new method.	Materials.
	Per cent.	*Per cent.*	*Per cent.*	
8285	2.72	3.25	0.53	Dissolved bone.
8281	2.17	2.75	.58	Mixed fertilizer.
8303	3.76	4.22	.46	Do.
8317	3.66	4.03	.37	Acid phosphate.
8265	3.15	3.84	.69	Mixed fertilizer.
8290	1.59	2.16	.57	Dissolved bone.
8286	2.96	3.85	.89	Acid phosphate.
8284	1.52	2.11	.59	Do.
8331	2.51	2.94	.43	Mixed fertilizer.
8333	.08	.31	.23	Do.
8332	2.92	3.39	.47	Do.
8310	.51	.95	.44	Do.
8309	.86	1.41	.55	Do.
8273	.33	1.00	.67	Do.
8276	.63	1.18	.55	Do.
8289	.04	.18	.14	Acid phosphate.
8287	.80	1.22	.42	Do.
Mean			.50	

Mr. Huston exhibited photographs of an apparatus designed to keep the fluid and solid in constant motion during the digestion for citrate-soluble phosphoric acid. The apparatus may also be used in work on feeding stuff and soil. The results so far obtained with it relate to insoluble phosphoric acid. In twelve cases reported the amount of insoluble phosphoric obtained was less than that obtained by the official method. Plates are necessary to understand the construction of the apparatus, and these can not be obtained in season for the publication of this bulletin. A full description will soon appear in a technical bulletin of the Indiana Experiment Station.

At the conclusion of these papers, there being no others on this subject to be presented, the presiding officer called for a discussion of the report on phosphoric acid.

The secretary called attention to the fact that a good day's work had been done and that the time for adjournment was near at hand, and suggested that the discussion of the subject of phosphoric acid be postponed until the morning session.

The presiding officer remarked that he thought also the time for adjournment had arrived. We have some latitude in regard to the time of adjournment under the order of the committee on order of business, but we have done enough, I think, to entitle us to a brief respite.

Mr. Van Slyke moved to adjourn until 9 o'clock to-morrow morning. The motion was carried and the meeting was declared adjourned at 5 p. m.

SECOND DAY.

FRIDAY—MORNING SESSION.

The chairman called the association to order at 9:30 a. m.

The report of the committee on potash recommendations was the first business called up.

Mr. PRESIDENT: Your committee reports its approval of the following recommendations and suggestions of reporters:

POTASH.[1]

1. On page 178 (4) insert "total potash in" before organic compounds.
2. On page 179, top line, change 10 to 30 minutes and 200 cc. to 300 cc.
3. On page 179, end of second paragraph, strike out "afterwards with 5 cc. of ether."
4. That a further investigation of the double decomposition between K_2PtCl_6 and the NH_4Cl wash be suggested for investigation by the reporter for 1895, and also further comparative tests of the Lindo-Gladding and Alternate methods in presence of the maximum amount of sulphates.
5. That in making up the solution in the Lindo-Gladding method in presence of soluble phosphate the use of $CaCl_2$, as suggested by Mr. Huston, be tried.

The recommendations were discussed seriatim.

Mr. ROSS asked if the committee considered recommendation No. 1 to apply to fertilizers containing organic matter. He supposed it included also fertilizers containing potash in organic compounds, or fertilizers containing organic matter, whether potash or not. Will all fertilizers containing organic matter, in order to get the total potash, have to be ignited in the manner prescribed?

Mr. WHEELER thought the difficulty comes from the fact that some States require the amount of soluble potash shall be determined; in that case the Lindo-Gladding method will be used. In other States the total amount of potash is called for, whether soluble or not, and the method of section 4 was adopted, as he understood it, simply to meet the demands.

Mr. BARTLETT did not see how one could misunderstand this paragraph or confound it with organic material in mixed fertilizers, because it says organic compounds, like tobacco stems, cotton-seed hulls, etc. Organic matter like bone phosphates, tankage, or matters of that character, are not at all like tobacco stems, so that it seems perfectly plain what is meant.

The PRESIDENT thought the difficulty might be obviated by changing the heading of paragraph (1) so as to include all mixed fertilizers;

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[1] All pages referred to in reports and discussions are in Bulletin 38, Division of Chemistry, unless otherwise noted.—H. W. W.

then the remaining paragraphs could be left as they stand. He imagined that heretofore we have understood section 1 "in superphosphates" to mean all mixed fertilizers, and section 4 to mean all those materials which are not mixed but which are evidently tobacco stems, cotton-seed hulls, and materials of that character. If there was any misunderstanding, paragraph 1 could be changed so as to include all mixed goods.

Mr. WHEELER. I infer there is objection on the part of a large number of members of this association to be forced to ignite mixed commercial fertilizers. If so, there might be a clause put in there saying, "not including mixed fertilizers" in the paragraph headed "organic compounds." That would make it perfectly clear to everybody.

Mr. FREAR. I think that where total potash is to be determined without any question, the last paragraph (section 4) could be modified so as to read in such a way as to make ignition required in all cases of mixed fertilizers in which total potash is to be determined, unless it is evident that the organic material present is not tobacco stems, cotton-seed hulls, etc. That makes ignition requisite wherever there can be any doubt as to the nature of the compounds furnishing potash.

Mr. HUSTON. I think the misunderstanding arises in the case of goods with, say, 4 or 5 per cent of available phosphoric acid derived from spent boneblack and enough castor or oil pomace of some sort to bring it up to 4 or 5 per cent of nitrogen, a very common mixture with us. The question is as to whether we shall ignite such goods. Shall we class them with superphosphates? If so, the ordinary worker puts them under section 1. Yet the bulk of such goods consists of castor pomace, and in such case with simple water treatment we get only about half the potash yielded after ignition. The question seems to be, How shall we classify the goods. It seems to me it is desirable to make some sort of provision for that sort of goods for the men who have to find total potash.

No further discussion arising, the president put the motion and the first recommendation was adopted.

The PRESIDENT. The second recommendation is to change the top line, page 179, to read, "boil for 30 minutes with 300 cc. of water."

Mr. WINTON. I think that some of this confusion could be avoided if, before methods for analysis, we had two or three methods for solution of phosphates. Of course, the method used for determining potash is independent of the method of solution. In reality both are the same so far as solution is concerned. We could in the first place have specific directions for making solution in the case of superphosphates, with special rules for bringing the material into solution for those States where they determine only water-soluble potash, and also rules for States where potash soluble in acid is to be determined. After that, give the methods for determining potash in these solutions. Paragraph 4 directs that the potash shall be determined by the Lindo-

Gladding method and does not allow the privilege of doing it by the Alternate method. It seems to me we should prescribe first how we are to make up the solutions and then how to analyze these solutions.

Mr. FREAR. I think the point well taken. I move that, so far as this matter of solutions is concerned, the recommendation be referred back to the committee with instructions to prepare such an arrangement of the method as shall separate prescriptions for solutions from those for determination of potash in the solutions.

Mr. WHEELER. I see no necessity for making the change. I think that at the bottom of page 179 it might read, "Proceed then as in the Lindo-Gladding or Alternate method" or "as specified by," etc., leaving everything else as it is. The question was brought up by the committee, who thought it would be taken for granted that it would refer to the first paragraph in either method. It is the intention to proceed either according to the Lindo-Gladding or Alternate method.

Mr. FREAR still believed the change would be wise; the arrangement of directions could hardly be too logical.

Mr. WINTON thought Mr. Frear's suggestion the only logical one to follow. In reading the paragraph the idea could not be got without considerable unnecessary attention.

The PRESIDENT. The amendment is to refer back the recommendation to the committee to arrange the order of preparation of the solutions, as I understand it.

The question was put and the amendment adopted.

The PRESIDENT. The third recommendation is, to strike out from the third paragraph on page 179 the words "afterwards with 5 cc. of ether."

Mr. WHEELER was rather opposed to striking out this clause. Washing with ether is erring on the safe side. He thought it would be better to put washing with ether into both methods rather than strike it out.

Mr. PAYNE thought that laboratories did not use ether now; they used alcohol. He had tried to see if it made any difference, and could not see any advantage in the use of ether. He always dried a quarter of an hour.

Mr. WHEELER. My idea was that in laboratory work there was danger of not drying long enough. With ether there is less danger of error. It seems to me it is erring on the safe side.

Mr. KILGORE thought washing with ether should not be made compulsory; it seemed not to be carried out by most laboratories. For that reason, and to make the methods the same in both cases as to washing, we preferred and voted to strike out the 5 cc. of ether.

The recommendation was voted upon and adopted.

The president requested the chairman of the committee to read again the fourth recommendation. This was done, to the effect that the suggestion be made to the reporter for 1895 to further investigate the double decomposition between the potassium platinochlorid and the common chlorid used in washing.

Also that further tests of the Lindo-Gladding and Alternate methods in the presence of maximum amounts of sulphates be made, and that in making solutions in the Lindo-Gladding method the use of $CaCl_2$ be tried.

Adopted.

Mr. McDonnell. I would like to move a further change, if it is in order at this point. I would move that under "Alternate method for potash," second line, 1,000 cc.; third line, 50 cc.; and fourth line, 100 cc., be replaced by half those amounts, respectively.

Mr. Huston. I suppose that goes to the committee of five on proposed changes in the methods; but I think it best to go a little slow in regard to that. These large volumes were taken purposely, and I think we would better make progress slowly in the direction of reducing the amount of these solutions.

The President. This, then, is referred to the committee on potash. We will now proceed to the report of the committee of five on soil and ash recommendations.

On motion it was decided to take up each recommendation and vote upon it separately.

CHANGES IN SOILS AND ASH METHODS OF ANALYSIS RECOMMENDED BY THE COMMITTEE.

1. Moisture to be determined in the air-dry soil by drying constant weight in a water over kept briskly boiling and all results of analysis to be referred to the soil in this dry condition and stated in the order recommended by the reporter of last year.
2. Digestion of soil to be made at the temperature of boiling water under atmospheric pressure for ten hours and with prevention of evaporation.
3. The hydrochloric solution to be analyzed essentially as in the appended method.
4. Five-tenths-millimeter sieve with circular mesh to be used in the preparation of soils.
5. That the study of a method for the determination of available P_2O_5 and K_2O in soils in lines similar to those by Dehérain, Dyer, and others and indicated also in the paper of Dr. Kastle before this association, is strongly recommended to the reporter for next year for investigation.
6. In ash analysis the soluble silica to be separated before ignition of the residue.

Mr. Payne. I would like to speak against the first recommendation. It seems to me that referring back everything upon the basis of the dry soil is really making the analysis on a basis in which the soil does not exist. To make an analysis of a peaty soil containing, say, 10, 40, or 50 per cent (of moisture) and refer it back to the dry basis, is certain to give the general public a very queer idea of the composition of the substance when they come to read it. When we make an analysis of that kind, the nearer we come to the actual facts, the better. Always to refer everything back to the dry basis really gives an incomplete idea of just what the soil actually is. The matter of soil analysis is bad enough now.

Mr. PETER. I think I can make it clear why the committee recommends this change. We want to refer the analyses, whatever they may be, to something definite and constant. We don't want to have our figures rendered uncertain, but for just the same reason that we refer the results of analyses of cattle feed to the substances dry at the temperature of boiling water, so we want to refer analyses of soils and all other products to some constant basis. It seems to us that the temperature of boiling water, as obtained under the working conditions of the laboratory, approaches more nearly a constant basis than anything we can obtain. This has been thoroughly settled in the case of fertilizers. It has been clearly shown that the results obtained in any other way are much less uniform than those obtained by drying.

Mr. PAYNE. Do you consider that that soil, after you have dried it for some time at 100°, is in the same condition as when you first took it up?

Mr. PETER. I am glad to see that Mr. Payne does not quite understand the recommendation of the committee. We do not mean to analyze the soil after it has been dried, but to determine the moisture so as to refer these results to something definite. We prepare our sample, get it into something like fair condition, bottle it, and find out how much moisture is there, and then weigh out portions and analyze them, referring the results obtained to this constant condition of the dry substance.

Mr. LINDSEY. On page 76 the reporter will notice that it is stated that the reason for adopting 200° instead of 100° is, that water will continue to come off indefinitely.

Mr. PETER. Yes, sir. If the association desires it, I will read some figures I read yesterday on that point. The fact is that these soils lose water continuously; sample No. 2 lost water after twenty hours' drying at 200°, but was reduced to constant weight in four to six hours' drying in the water oven. I found this to be the case in the majority of soils. The loss of weight on drying in a water oven practically ceased in a reasonable time.

Mr. DE ROODE. I can corroborate the statement of Mr. Peter, so far as drying at 200° is concerned; I rarely ever get a constant weight.

No further discussion arising, the recommendation was voted upon and adopted.

The second recommendation was read by Mr. Peter.

Mr. DE ROODE. I think it ought to be a little more definitely stated how to prevent evaporation. To say a Schneider flask would be a little more explicit.

Mr. LINDSEY. We have been talking of digesting for five days. That seems a long time to digest soil, but from that to ten hours is a pretty long stride. It seems to me before that be adopted it ought to be looked into for another year.

Mr. **Peter.** The limit of time was not insisted upon by the reporter as much as other conditions. The limit of time, in my opinion, should be regulated so that the results will agree as nearly as possible with the results of the beaker method, which is practically the method that has been practiced in this country heretofore, so as to keep our future results as nearly in agreement with these as possible. I simply suggested ten hours as a compromise between the recommendation of two hours made in 1892 and the time set in the Provisional method, thirty-six hours.

The recommendation was voted upon and adopted.

The third recommendation was read by Mr. Peter and was adopted.

The **Secretary.** I understand this is no longer a Provisional method; it is a method?

The **President.** Yes, sir.

The fourth recommendation was then read.

The **President.** This is essentially a suggestion to the incoming reporter.

Mr. **de Roode** wished to know if the suggestion compelled the reporter for next year to take up that line of work.

The **President** thought he could not be compelled to do so.

Mr. **Wheeler.** I think the idea is that this is a very important line of work never taken up in this country, and more promising than the regular soil analysis. It is simply a suggestion, so as to call it to his attention.

The **President.** No doubt a very good suggestion.

Adopted.

The fifth recommendation was then read.

Mr. **Payne.** The heading "Preparation of ash," page 205, seems to me rather misleading. It says "Before combustion the material must be thoroughly cleaned from all foreign matter, especially adhering soil, wood, bark, roots, etc." As I understand it, this is a description of how to make an analysis of a plant. If we remove all adhering substances there would not be much left. It is very confusing as it is now.

Mr. **Wheeler.** I would like to accept the gentleman's amendment and strike out the words "wood, bark, roots, etc.," making it read "the material must be thoroughly cleaned of all foreign matter, especially soil."

The **President.** Do you include this in the fifth recommendation?

Mr. **Peter.** Yes, sir.

The recommendation was then read as amended, and adopted.

The sixth recommendation was then read, to the effect that a sieve with a mesh of one-half millimeter be used in the preparation of fine earth for analysis.

Mr. **Huston** thought this was a matter that should be specified more accurately. He thought no more important matter could come before the association than definitely settling the size of the orifice through which this material is passed before we begin work upon it.

Mr. **Peter** said he would much prefer to define the sieve as one with a circular opening one-half millimeter in diameter, but there was some objection to that kind of sieve, and he thought it best not to define it.

The President then called for any further papers on the subject of phosphoric acid in continuation of the business of last evening.

Mr. **Huston**. I have something which is, perhaps, between a formal paper and a discussion. It is merely an apparatus which I have had an opportunity of testing only a few times, which I believe has a bearing on cattle foods as well as soils. It is a means of better mechanical agitation in making solutions.

(Mr. Huston here exhibited photographs of his apparatus and said he would write up his remarks for the secretary.)

The **President**. Are there any further papers to be presented?

Mr. **Terne** wished to thank the reporter (Mr. Kilgore) for including in his report the results of work in his (the speaker's) laboratory. He felt they were indebted, as members of the Franklin Institute, to have the results of their work taken up so quickly. The results they had achieved in his laboratory were very encouraging, and Mr. Kilgore had included in his report the results of the work of Mr. Bergami, his assistant. The main result was that we got somewhat lower results than with the Official method, but the differences were not any greater, but were less, than the differences you gentlemen have reported. The saving of time which is achieved by the titration method I think is worth a great deal to those who have to do with phosphoric acid samples in large numbers, and I really think the method is just as good in the hands of a skillful chemist as the Official method. If you conclude to give this method another year's trial, it would be in the interest of great advancement in our methods; and it would be a very good check to have two methods for our phosphoric acid determination; the Volumetric method to check the Gravimetric method, or *vice versa*.

Mr. **Ross**. I have a few words to say with regard to the Direct method for the determination of citrate-soluble phosphoric acid. The method I proposed had the misfortune, although that seemed to be the only basis of comparison, to be compared with the citrate-soluble obtained by difference. It is a well-known fact that citrate-soluble by difference, like nitrogen per extract, is the dumping ground for all errors in all other determinations. I did not myself expect to find the closest agreement between the method I proposed and the results which were secured by difference. To show the errors that are liable to arise by such a comparison, if we look at the figures on insoluble in sample No. 1, secured by regular association methods, we shall find that these results vary all the way from 0.03 to 0.45; the mean is 0.27, the greatest variation is 0.42, or a variation of 150 per cent. So that under these circumstances, although the showing against this method is rather unfavorable, yet the figures on which this comparison is based possibly can not be considered the most reliable in the world, considering that the errors from the other determinations will certainly creep into it. The peculiar part about some of the work on this subject, which I do not understand (quite a number said they had trouble on this question), is that this sulphuric-acid method will apply to total phosphoric acid in mixed fertilizers of every kind where organic matter is in an insoluble condition and offers great resistance to the action of the oxidizing agents. We have also incorporated it in our methods for determining insoluble; but when it comes to destroying the citric acid which is already in solution, and which, if the method is properly performed, can certainly be destroyed in twenty minutes' digestion if worked with care, it seems the results do not agree, which is something I am unable to understand. Possibly some details of the execution of the method have been overlooked by some, or I have failed to state explicitly enough in my description of the matter. One point to which possibly I did not call sufficient attention is, that the full amount of ammonia citrate prescribed must be used and the digestion must be carried on the full length of time required. I made some experiments with chemically pure phosphates, mixing with them some ammonium-citrate solution, and then went through digestion in the regular manner. I had no trouble in securing satisfactory results. It is true that the large amount of ammonia citrate used makes the precipitation of the ammonium phosphate liberated rather slow, but tests I made showed that the liberation was certainly complete even before the time of digestion was up. I fail to understand why this method, which involves the same principle in the determination of phosphoric acid in organic samples of every kind, fails to give good results when it is applied to solutions containing organic matter or in liquid condition and in such form that it can be readily destroyed.

Mr. Wheeler. I would like to ask if the digestion was continued the right length of time in all cases?

Mr. Ross. I am not posted as to the length of time the digestion was continued. I have had no trouble where the digestion was continued the right length of time.

Mr. Kilgore. Mr. Wheeler means digestion with sulphuric acid. I should think there would be no trouble there, because the directions for making the determination specify to digest until the liquid is clear. With reference to the method of Prof. Ross for the direct determination of citrate-soluble, in the hurry I was necessarily in in getting together the immense mass of matter that composed my report, I hardly think I did the subject justice, because I am really in sympathy with the spirit of the method, but the results would not allow me to lean either one way or the other. I felt I must report results just as they were. I would say, also, that the committee are of the same opinion that I am, that it is a step in the right direction; that if it would work it would eliminate some sources of error. When further washing with water is continued with acid phosphates, especially every time a fresh portion of water is added, it will run through cloudy. The Ross method, if it gave good results, would eliminate this error and, I think, give more uniform results for available phosphoric acid.

Mr. PAYNE. In regard to this matter of phosphates running through, with the soft phosphates of Florida I find that often a lot of substance runs through the filter, so that you can not filter it all. In many cases this is given as reverted phosphoric acid, but in several cases I allowed some of this filtrate to stand until it finally settled, then decanted the water and determined the amount of total phosphoric acid in this sediment. In one case it amounted to as much as 0.5 per cent, simply on account of the minute condition of the soft phosphate. I know we always got a little more than it is entitled to.

CHANGES RECOMMENDED BY THE COMMITTEE IN METHODS OF ANALYSES FOR PHOSPHORIC ACID.

1. That on page 169, sixth line from the bottom, after the word "dissolved" add "or return the filter with contents to the digestion flask, add 30 to 35 cc. strong nitric acid and 5 to 10 cc. strong hydrochloric acid, and boil for not less than forty-five minutes."
2. That on page 170, second paragraph under "total phosphoric acid," next to last line, insert after filter "wash with 2½ per cent NH_3 solution until free of chlorids, dry and ignite at first over a low flame, finally over the blast lamp, until the precipitate is perfectly white, and weigh."
3. That on page 171 the directions for making dilute ammonia wash be made to read: This solution shall be prepared so as to contain 2½ per cent NH_3.
4. That the Volumetric method for phosphoric acid by titrating the yellow precipitate and also the method of Ross for citrate-soluble phosphoric acid be made subjects of further study.

Mr. WHEELER. I would like to add my testimony to Mr. Kilgore's. This association has been working for many years on insoluble phosphoric acid and getting the available by difference. When Mr. Ross proposed his method it seemed to me he had struck just the right thing. What we want to arrive at is the citrate-insoluble, and if we can get a direct method for this we shall get much more uniform results than we are getting now. I think the association ought to investigate the matter further.

The PRESIDENT. If there is no further discussion it is in order to refer this report to the committee.

Mr. KILGORE. I would state that the recommendations of the reporter were read before the association yesterday afternoon and the committee acted on these recommendations last night.

The PRESIDENT. We will then take the report of the committee on phosphoric acid and recommendations.

Moved and seconded that the recommendations be considered separately. Adopted.

The first recommendation was then laid before the association.

Mr. MCDONNELL. I am in favor of part of that amendment, but it seems to me digestion with acid is continued longer than necessary, at least in most cases. Forty-five minutes is a very long digestion.

Mr. **Wheeler**. That is a matter which has not been carefully worked up. I think it was the opinion of the committee that it was better to set the time long enough now in adopting it than to set it a little short, as long as the matter has not been studied.

Mr. **de Roode**. I see no necessity to set the time at all. State it, digest until all the phosphate is dissolved.

Mr. **Kilgore**. I would say in support of this that in the experience we have had with the method which has been used continuously in our laboratory for five years, in the case of phosphates and materials of that kind, they go into solution as quick as, if not quicker, than total phosphoric acid; but for fear of not getting all fish scrap, tankage, and material of that kind, we always boil for forty-five minutes. On all other materials the time for phosphoric acid is perfectly safe; we merely do this to be sure.

Mr. **McDonnell**. I would like to offer as an amendment that the words "until all the phosphate be dissolved" be inserted instead of "forty-five minutes," the same as under total.

Mr. **Kilgore**. I could not accept that without the consent of the committee, as it was put in by the committee.

Mr. **Peter**. How are we to tell when the phosphate is dissolved? The length of time has been determined by investigation; the reporter for this year has determined the time in which the phosphoric acid is surely dissolved in all his experiments, and he tells us what his results are. Why go back of this and leave it to the individual to do the work over again?

Mr. **Huston**. I see a little difficulty in boiling forty-five minutes. At the rate we boil in our laboratory the flask would be dry in thirty minutes, unless we can add additional acid if necessary. I believe if we undertake to follow this method some workmen with short-necked flasks would get into difficulties.

Mr. **Peter**. We are not so rapid in our laboratory as Mr. Huston is, but I can not see that you get the phosphate in solution any faster by boiling rapidly than by boiling slowly; the temperature is the same.

Mr. **Payne**. Evaporation is very much more rapid in a strong draft and it draws the acid out of the flask in double time.

Mr. **McDonnell** failed to see why the time was so much more important here than under total phosphoric acid.

Mr. **Frear**. I wish to emphasize this point. We are perfectly safe in leaving it to the individual analyst. I doubt whether we should be prepared to specify the precise time limits to hold in all cases, because it has been very clearly indicated that the condition of the solution as regards rate of evaporation does undoubtedly vary to some extent. The material lies in the bottom of your flask, and while it may seem that on top the same density exists, I am quite confident that at the bottom, where the material lies, there is some difference of temperature.

The **President**. On these questions of official work I suggest we have a rising vote. Only those charged by law with official control of fertilizers have the right to vote on these questions.

Eight rose in favor of the amendment, 3 against it.

The PRESIDENT. Adopted. The question is now on the original recommendation as amended.

Thirteen rose for, none against.

Adopted.

The second recommendation, page 170, was then read.

Mr. DE ROODE. Instead of NH_3 I would substitute the word "ammonia."

Mr. KILGORE. We put it NH_3 because in making solutions for washing it would be done from specific gravity, and all our tables of specific gravities give the per cent of ammonia in terms of NH_3, and 2½ per cent is the usual strength used.

Mr. PAYNE. Ignition to whiteness is very difficult to secure sometimes, and if the ignition is thorough it certainly does not make any difference, as far as we have been able to find out.

The PRESIDENT. Was the reporter able to get his precipitates perfectly white?

Mr. KILGORE. I never have any trouble, when they are thoroughly washed and dried and burned properly. Sometimes it is grayish white, and it never should be anything but white or grayish white material. I would say we aimed at perfection, and want everybody to get as near that as possible.

Mr. FREAR. It seems to me that the committee in doing control work and aiming at perfection is making a mistake. What we want is the closest result practically attainable. There is no doubt that a very little bit of matter might be burned off, producing a somewhat whiter precipitate than at first obtained, and yet when we come to weigh it, it would have no appreciable result on the commercial value of the goods we were testing. I have no objection to any amount of precaution requisite to secure thoroughly reliable work up to the degree required by commercial conditions.

Mr. PAYNE. I worked on that some years ago. Fresenius does not seem to think it necessary for it to be absolutely white. I ignited a lot of my phosphates, probably several hundred determinations, over a very strong Bunsen, and got them in very nice condition without any great trouble, using platinum crucibles. When I used a blast lamp I had six platinum crucibles ruined in about six days; they all went to pieces.

Mr. PETER. In my experience I found that when the precipitates are ignited with carbon in the platinum crucibles they will injure the crucible, but when carbon is burned at low temperature no injury at all arises. It seems to me that the instructions if followed out will lead to white precipitates.

Mr. DE ROODE. That has been my experience. We have had no trouble at all in getting perfectly white precipitates. I think it would look better to have our methods described in a correct way, and avoid any criticism from outsiders.

Mr. Wheeler. I will state that our methods have been criticised by European writers in the matter of washing. We do not specify that they should be washed until the filtrate gives no precipitate. We should specify in such a way that the test would be an accurate test.

Mr. Payne. I think Mr. Wheeler's remarks are very pertinent indeed. There are a great many chemists, I find, who do not test wash water to see that they are thoroughly rid of the substance they are trying to get rid of. Some persons seem to be able to wash material perfectly free with half the wash water that others use, and yet get a better precipitate. I think it is very well in all our methods that the filtrate should be tested until it is free from the substance we are trying to get rid of.

Mr. Frear. We are not devising methods for the determination of atomic weights. We are devising methods which are to be applied to certain commercial problems, and any time or money spent on refinements of methods beyond the requirements placed upon us is wasted. We teach nobody anything; we do no possible good in the world by making fertilizer analyses with exceptional refinement. If the method were designed to accomplish any other purpose, then overrefinement should be introduced, but now I hold that only those precautions should be taken which are necessary to prevent errors which must not enter into such analyses as we are making. Any further refinement is a waste.

Mr. Kilgore. If we allow inaccuracies to get in, as from incomplete washing, then, under our methods, we are obliged to accept them; while, if we have the methods which must give right results, it does not matter if results are wrong, for only the analysts are responsible for them. One trouble spoken of by Dr. Payne of its taking more wash liquid with one man to wash precipitates free of chlorids than with another, is due very largely to the amount of magnesia mixture employed in precipitation. Some of the workers tell me they use 20 cc., which is, perhaps, twice what they should use. I have found that the more magnesia mixture used the more washing it takes. As to ruining your crucibles by burning to whiteness, we have no trouble in our laboratory. By regulating the heat we have no difficulty whatever.

Mr. Bartlett thought the method used in filtering had much influence on the result.

Mr. Finney considered this one of the most important points brought before the association for some years. It made a difference whether the burning was to white or gray. He had found a difference of 0.02 to 0.03 per cent. With regard to the recommendation by the reporter, he could see no use in washing free from chlorids. Magnesia is the only thing we have to get rid of, and in his laboratory he used a solution of phosphate, testing for the magnesia, and when washed free from magnesia he got accurate results.

Mr. McDonnell. What do you mean exactly by free from chlorids? We should say, I think, "until the chlorids are practically removed," or something of that sort.

The question on adopting the amendment was put to a rising vote by the president. Six voted for, 7 against; defeated.

The third recommendation was then read and adopted.

The fourth recommendation was then read and adopted without discussion.

Mr. Ross called attention to a chart which he used in his laboratory for obtaining percentages of phosphoric acid without making calculations and without reference to tables.

The president then called for the report of the reporter on sugar.

Mr. Spencer read the following report:

REPORT ON SUGAR.

By G. L. Spencer.

The reporter on sugar, by direction of the association, has continued the work on the determination of moisture. As is usual in sugarhouses the percentages are expressed in terms of the total solids.

The accurate estimation of the moisture in the class of materials with which the sugar chemist must deal is a most vexatious problem, and one upon which much labor has been expended, both in this country and abroad, and which is still without a solution.

Three association chemists and two refinery chemists coöperated with the reporter in work. As little work was asked of the analysts as was necessary to securing the required data. Five chemists applied for samples and failed to report.

I am indebted to Dr. F. G. Wiechman, of Columbia College and the American Sugar Refining Company, New York, for a very complete report, and to Mr. L. W. Pickart, assistant chemist in the Standard Refinery, Boston, who, through the courtesy of the chemist, Mr. L. J. Schiller, made as many determinations as his refinery duties would permit.

The following suggestions were made to the various chemists by the reporter:

1. Follow the Official method as described in Bulletin No. 38, p. 179, using about 1 gram of the material. Report the total solids as indicated by each weighing.

2. Heat about 1 gram of the substance in some convenient form of vacuum apparatus; report the total solids as indicated by the weighings after five, ten, fifteen, and twenty hours' drying. If you can spare the time continue the heating for about forty hours. The temperature of the air or liquid surrounding the containing vessel to be maintained at approximately 100° C.

3. The paper coil or so-called Jossé method. Transfer about 2 grams of the substance to a small round dish, flat bottom; dissolve in 6 to 8 cc. water, applying heat if necessary; absorb the liquid on a coil of paper made from the paper you will find inclosed in the package with the samples. Dry two hours and weigh; dry during three or four additional intervals of two hours and report the total solids indicated by each weighing. The temperature of the oven should be approximately 105°. It is obvious that precautions must be taken to prevent absorption of moisture by the coil of paper while weighing.

Two samples were sent the analysts: No. 1 was sorghum molasses. No. 2 consisted of diabetine (commercial levulose) and cane sugar, 40 per cent of the solids being diabetine.

Sorghum molasses was selected, since it probably contains the impurities which render the drying of similar substances so uncertain. Levulose was added to sample No. 2 on account of the low temperature at which it is said to undergo a change, and further because it is naturally present in cane molasses.

Assuming that the cane sugar and levulose have practically the same specific gravity, the composition of sample No. 2 is such as to permit a calculation of its total solids from data obtained by dilution and pyknometer. Messrs. T. F. Sanborn and Oma Carr, of the Chemical Division, independently of one another, estimated the total solids by this method. The results were respectively 74.51 and 74.48 (mean 74.50) per cent. In one case several times as much of the material was taken as in the other. The close agreement of the calculated solids, in the two estimations, indicates that no inorganic matter was present and that the sample consisted only of the carbohydrates in question and water; I believe under the conditions, that this sample contained very nearly 74.50 per cent solid matter.

Owing to the fact that there is some decomposition of levulose between 50° and 60° and complete decomposition at 100°, with formation of volatile products at the latter temperature, the specific gravity method is the only one available for a reasonably accurate estimation of the solids in sample No. 2.

Total solids in samples supplied by reporter on sugar.

SAMPLE No. 1.

Official method.					Paper-coil method (Jossé).					Vacuum method.			
Hours.	Prof. Scovell.	Dr. Wiechman.	Prof. Heileman.	Mr. Carman.	Hours.	Prof. Scovell.	Dr. Wiechman.	Prof. Heileman.	Mr. Carman.	Hours.	Prof. Scovell.	Dr. Wiechman.	Mr. Carman.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*
10	76.62	75.07	76.15	75.23	2	73.79	74.03	75.73	75.50	5	78.92	75.28	75.73
11	76.41	74.92	......	75.12	4	73.41	74.50	73.75	74.47	10	77.13	75.15	74.90
12	76.25	......	......	74.95	6	73.10	73.70	[2]73.25	73.64	15	76.54	74.97	74.87
13	......	......	75.87	74.94	8	73.09	73.48	......	73.63	20	75.22	74.94	74.49
14	76.21	......	75.82	74.83	10	72.68	......	......	73.57	25	75.13	74.75	74.48
15	......	......	......	74.78	12	72.58	......	......	72.88	30	74.52	74.67	74.24
16	75.22	......	[1]75.53	74.73	14	72.34	......	......	......	35	73.64	74.65	74.06
17	75.04	......	......	......	16	72.44	......	......	......	40	......	74.58	74.05
18	74.90	......	......	......	18	72.14	......	......	......	....	......	......	......
20	74.70	......	......	......	....	......	......	......	......	....	......	......	......
22	74.60	......	......	......	....	......	......	......	......	....	......	......	......

[1] On a second trial Prof. Heileman obtained the following per cents at ten, twelve, and thirteen hours, respectively: 75.56, 75.46, 75.36.

[2] Six hours.

Open-dish, Mr. Pickert.[1]			Open-dish work repeated.		
Hours.	At 105°.	At 105°.	Hours.	At 105°.	At 105°.
	Per ct.	*Per ct.*		*Per ct.*	*Per ct.*
3	76.53	75.30	3	75.49	75.63
5	76.28	75.02	5	75.36	74.59
7	75.50	74.61	7	75.26	75.44
9	75.01	......	......	......	......

[1] 2.5 grams with 30 grams of sand, first dried two hours at 95°, then at 105°.

SAMPLE No. 2.

Official method.					Paper-coil method (Jossé).					Vacuum method.			
Hours.	Prof. Scovell.	Dr. Wiechman.	Prof. Heileman.	Mr. Carman.	Hours.	Prof. Scovell.	Dr. Wiechman.	Prof. Heileman.	Mr. Carman.	Hours.	Prof. Scovell.	Dr. Wiechman.	Mr. Carman.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*
10	77.19	75.52	74.70	74.93	2	74.80	73.35	73.77	73.67	5	77.27	74.95	74.10
11	77.06	75.47	......	74.83	4	74.45	72.87	73.75	73.04	10	76.07	73.95	73.83
12	76.89	......	......	74.70	6	74.22	72.85	73.10	72.16	15	75.57	73.80	73.62
13	......	......	[1]74.36	74.69	8	74.02	72.73	......	72.20	20	74.73	73.71	73.30
14	76.73	......	74.36	74.59	10	73.71	......	......	72.15	25	74.56	73.57	73.19
15	......	......	......	74.59	12	73.63	......	......	71.42	30	74.41	73.40	72.98
16	75.79	......	74.22	74.53	14	73.38	......	......	......	35	73.29	73.33	72.55
17	75.67	......	......	......	16	73.31	......	......	......	40	......	73.38	72.40
18	75.47	......	74.22	......	18	73.08	......	......	......	....	......	......	......
20	75.26	......	......	......	....	......	......	......	......	....	......	......	......
22	75.12	......	......	......	....	......	......	......	......	....	......	......	......

[1] On a second trial Prof. Heileman obtained the following per cents at 10, 12, and 13 hours, respectively: 74.54, 74.24, 74.20.

Open dish (Mr. Pickert).[1]				Open-dish work repeated (Mr. Pickert).		
Hours.	At 105°.	At 105°.	At 105°.	Hours.	At 105°.	At 105°.
	Per ct.	*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*
3	74.17	74.66	74.21	3	74.50	74.45
5	74.14	74.27	74.18	5	74.41	74.39
7	74.06	74.01	....	7	74.33	74.35
9	73.76	....	....	....	....	....

[1] 2.5 grams with 30 grams of sand, dried first at 95° two hours, then at 105° C.

Summary of results obtained on samples Nos. 1 and 2; total solids by various methods.

Prof. Scovell.			Dr. Wiechman.			Prof. Heileman.			Mr. Carman.		
Official.	Paper-coil.	Vacuum.	Official.	Paper-coil.	Vacuum.	Official.	Paper-coil.	Vacuum.	Official.	Paper-coil.	Vacuum.
Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
76.25	73.09	75.13	74.92	74.00	75.15	75.82	[1]73.25	....	75.12	73.63	74.87
77.06	74.02	74.56	75.47	72.85	73.80	74.36	[1]73.75	....	74.83	72.15	73.19

[1] Drying not completed.

In sheet No. 2 of the tabulation is given a brief summary of the results obtained in the experiments. With sample No. 1 the maximum per cent solids obtained was 76.25 and the minimum 73.25, a difference of 3 points. The results are not at all uniform. Unfortunately it is impossible to say which of these determinations shows more nearly the correct percentage. With sample No. 2 all three methods give results closely approximating the correct one, but not in the hands of one chemist. The maximum is 77.06 and the minimum 72.15.

In selecting the final weighing upon which to base the percentages, the rule laid down in the Official method was followed to a certain extent, viz, continue drying until the loss of water is not greater than 0.20 per cent in 1 hour. The exception to this rule was in the substitution of the periods given in the tables, in the statements of the work by the vacuum and paper-coil methods, for the period of 1 hour required by the Official method.

The results obtained by Mr. Pickert on sample No. 2 agree quite closely with theory.

CRITICISM OF METHODS.

The rate of drying by the paper-coil method is extremely rapid. A coloration appears at an early stage of the drying, indicating a decomposition. It is difficult to thoroughly dry the paper. Paper will sometimes continue losing weight for many hours. The results obtained by this method appear to be unreliable. This may be largely due to the fact that most drying ovens are of such construction that in no two parts can the same temperature be obtained. Many of the irregularities are very probably chargeable to the oven rather than the process. Drying should not be long continued in working by this method, since after the fourth hour the loss in weight is probably not due to the evaporation of water.

The vacuum method is open to the serious objection that entrainment is very liable to occur. The time required for drying is excessive. The results are irregular.

There is nothing in the data obtained in these experiments to lead one to believe that the other methods tried are more certain of yielding satisfactory results than the official method. With a perfectly uniform temperature and properly ventilated ovens it is probable that the official and paper-coil methods would have yielded more uniform results.

RECOMMENDATION.

The results of these experiments do not justify the reporter in making recommendations for changes in the method now official, or for the addition of other methods. I recommend, however, that the reporter for next year continue the investigation of methods of moisture determination.

ESTIMATION OF THE DRY SUBSTANCE IN SAMPLES OF SUGAR SENT TO DR. FRIEDRICH STROHMER.[1]

The dry substance in samples 1 and 2 was determined by the methods sent with the samples and by drying to a constant weight in a vacuum oven. The method by drying on paper rolls was not tried because the rolls necessary to its conduct were not sent.

	Dry matter.	
	Sugar 1.	Sugar 2.
OFFICIAL METHOD.	*Per cent.*	*Per cent.*
After 16 hours' drying	74.95	74.51
After 20 hours' drying	74.78	74.20
DRYING IN A VACUUM OVEN.		
After 4 hours' drying	75.92	75.58
After 8 hours' drying	75.53	75.12
After 12 hours' drying	75.21	74.78

After twenty hours' drying by the official method the weight remained constant. After twelve hours' drying by the vacuum method the weight remained constant. The difference between the two methods in sugar No. 1 amounted to 0.57 per cent, and in the second sugar 0.58 per cent. It is seen that the constant weight was reached in a vacuum oven in about half the time required by the drying in an open dish.

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[1] Received too late for incorporation in Mr. Spencer's report.

After some discussion, there being no recommendations in the report, the president decided that the report was before the meeting for adoption. It was received and adopted, and the president called for further papers or discussions under this head.

The secretary presented the following paper by Messrs. Weber and McPherson:

ON THE DETERMINATION OF CANE SUGAR IN THE PRESENCE OF COMMERCIAL GLUCOSE.

By H. A. Weber and William McPherson.

In the analysis of a great many of the saccharin products found in the markets at the present time, the chemist is confronted with the problem of determining sucrose in the presence of commercial glucose. The Official methods for such determinations, either by the use of Fehling's solution alone or supplemented by the polariscope, necessitate, as is well known, the inversion of the sucrose by the action of acids. The accuracy of these methods requires, as one condition, that the acid used in inversion exert no action upon any substance present, other than sucrose, that would in any way affect the reading of the polariscope or would change the power to reduce Fehling's solution. Since commercial glucose, however, contains a greater or less amount of dextrin, and since this undergoes hydrolysis when acted upon by acids, being changed thereby into dextrose, the question arose as to whether such a change might take place during the process of inversion of any sucrose present and thus vitiate the results. The specific rotatory power of dextrin being nearly four times as great as that of dextrose, it is evident that a very slight action would introduce serious errors.

The method of procedure was, first, to determine to what extent dextrin is present in commercial glucose; second, to ascertain whether any change is produced by heating with acids as in the common process of inversion, and finally to discover some method of eliminating this error, if present.

An examination of the literature accessible upon the subject of the composition of American glucose failed to give any information in regard to the amount of dextrin present.

The report on glucose, Washington, 1884, gives the analysis of some glucoses, but, as suggested by Stone and Dickson improvement or variation in the manufacture since that time would probably cause a change in the chemical composition.

In order to obtain the desired information a number of samples were analyzed. Seven of the samples were kindly sent us by the American Sugar Refining Company, of Chicago, and represent the various goods placed on the market by them. Since this company controls the glucose manufacture in the United States, it is fair to presume that these samples represent, more or less fairly, an average glucose in the market. Two other samples were purchased in Columbus, but analysis showed a composition similar to those sent from Chicago, and inquiry revealed the fact that they had been purchased from the above-mentioned company.

METHOD OF ANALYSIS.

A number of different methods have been proposed for the examination of the constituents of glucose, all more or less objectionable. The shorter ones are only fairly approximate, making no attempt to estimate the amount of the so-called inert or unfermentable carbohydrates (gallisin). The longer methods are probably more exact, but, since the unfermentable carbohydrates are but imperfectly known, it follows that any attempt at finding the exact composition can not be highly satisfactory.

The following method[1] was selected as giving approximately the relative amounts of dextrose, maltose, and dextrin present.

Twenty-five grams of the glucose were dissolved in water, and the solution made up to 250 cc. With this solution the following determinations were made:

1. Specific gravity.—This was determined by weighing with the pyknometer. The Westphal balance was first used, but the results were not so satisfactory as those obtained by direct weighing.

2. Specific rotatory power.—This was deduced from the direct reading in a 200 mm. tube. The instrument used was the ordinary Schmidt & Haensch polariscope. Its accuracy was tested by a standard plate, kindly loaned by Dr. Wiley.

3. Cupric reducing power.—A gravimetrical determination was made by adding 10 cc. of the solution properly diluted to 40 cc. of Fehling's solution previously heated in boiling water until it attained a like temperature and then heating for twelve minutes in water at 100°. The cuprous oxid was filtered through asbestos, well washed, and changed into the cupric form by igniting for fifteen minutes. The weight of the oxid multiplied by 0.4535 gives the amount of reducing sugar present. Experiment showed that good results could be obtained by exercising due care and adhering rigidly to the conditions.

4. Ash.—Fifty cubic centimeters of the solution were treated with sulphuric acid in a platinum dish until all the carbonaceous matter was burned away, the residue weighed, and the percentage of ash calculated.

The total organic solid matters per 100 cc. were determined by subtracting from the specific gravity of the solution (water=1,000) the correction due to the ash, and dividing the difference between the result and 1,000 by 3.86. This is based on the assumption that 10 grams of carbohydrates dissolved in 100 cc. of water give a solution of a sp. gr. 1,038.6 (water=1,000), also that 1 gram of ash per 100 cc. gives a specific gravity of 1,008. Now, if in a solution containing 10 grams. to 100 cc.,

S =total carbohydrates.
$(A)d$=specific rotatory power for, say, D.
K =cupric reducing power in terms of dextrose,

Then

$$\text{Maltose (M)} = \left(\frac{(A)d+1.42\ K-195)\ S}{27.2}\right)$$

$$\text{Dextrose (D)} = \frac{SK}{100} - .61\ M$$

$$\text{Dextrin} = S-(M+D).$$

It is evident that this method will give only approximate results, since it supposes that the only organic bodies present are maltose, dextrose, and dextrin. However, the results undoubtedly give a fair insight into the amounts of these present.

TABLE NO I.—*Glucoses analyzed.*

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Specific gravity	1030.60	1031.05	1031.40	1031.18	1033.16
Total solids, grams	7.8890	8.01	8.08	8.03	8.56
Specific rotatory power	133.33	132.9	120.6	135.4	132.1
Ash, per cent	.18	.19	.24	.21	.17
Cupric reducing power, per cent	56.56	57	60.60	54.9	56.7
Maltose, per cent	54.08	55.44	34.63	54.20	55.4
Dextrose, per cent	11.64	11.81	21.24	11.03	14.71
Dextrin, per cent	13.18	12.80	24.98	15.09	15.44

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[1] See article Sugar, Thorpe's Dictionary of Applied Chemistry.

TABLE NO. I.—*Glucoses analyzed*—Continued.

	No. 6.	No. 7.	No. 8.	No. 9.	No. 10.
Specific gravity	1033.90	1031.17	1031.48	1033.50	....
Total solids, grams	8.74	8	8.11	8.60	....
Specific rotatory power	127.8	149.3	130.31	134.91	40.2
Ash, per cent	.22	.37	.29	.37	....
Cupric reducing power, per cent	55.9	43.6	50.65	48.75	....
Maltose, per cent	39.12	47.67	22.30	28.92	....
Dextrose, per cent	25.08	5.81	27.48	24.31	....
Dextrin, per cent	23.18	26.52	31.33	32.81	....

It is of interest to compare these results with the following, the first three of which are taken from the report on glucose above mentioned. The fourth is the analysis published recently of a product manufactured in England.

	No. 1.	No. 2.	No. 3.	No. 4.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*
Maltose	19.3	7.6	....	36.10
Dextrose	36.5	36.5	39	18.75
Dextrin	29.8	40.9	41.4	25.41

The most noticeable feature of the above analyses is the high per cent of maltose present and the comparatively low per cent of dextrose. With a single exception the maltose present exceeds the dextrose, reaching a maximum difference in number 7, which contains 47.67 per cent maltose and only 5.81 per cent of dextrose. This particular sample was labelled "Brewers' Extract," the name suggesting a high per cent of maltose. A characteristic property of the glucoses obtained from Chicago was a malt-like odor noticeable when dissolved in a slight amount of hot water. It will be noticed that the English sample contains almost twice as much maltose as dextrose.

The solid glucose or grape sugar was not analyzed completely, since the specific rotatory power was so low as to exclude any possibility of the presence of dextrin.

It is of interest to compare the properties of the above glucoses with those examined by Stone and Dickson.[1] The determinations made by them were, in the main, different from those made by us, overlapping, however, in the determination of the specific rotatory power and ash. A marked difference exists in these. The average specific rotatory power of those examined by Stone and Dickson is given as 91.37, while those examined by us showed a variation from 120.6 to 149.3, calculated for ray D. It is possible that the former calculations may have been made for some other ray, and the apparent difference thus diminished. The percentage of ash in the samples examined by us was noticeably smaller, being about one-fourth as great.

The glucoses were next subjected to the same treatment as would be necessary to invert sucrose if present, viz, heating with acids. It is evident that in order to guard against any hydrolysis of the dextrin or to diminish it, if unavoidable, the temperature to which the acid solution is raised as well as the time during which this temperature is maintained should be no greater than necessary to insure complete inversion of the sucrose. Various methods of inversion have been proposed and various ones are in use by chemists at the present time. The Official methods of the agricultural chemists for the years 1890 and 1891 require that the solution to be inverted, mixed with the proper amount of acid, be heated for ten minutes at 68°. This was changed in the report for 1892 and continued in the report for 1893, to heating gradually, reaching 68° at the expiration of ten minutes. This is also the method given by Allen,[1] as well as the one recommended by the "Association des Chimistes."[2]

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[1] See this journal, Vol. VII, No 6.

Three different methods were tried: 1. The above-mentioned or Official method; 2. The method recommended by the Association of Official Agricultural Chemists for the years 1890 and 1891, viz, heating at 68° for ten minutes; 3. The German method, which amounts to heating at 67° to 70° for fifteen minutes.

Approximately 26.048 grams of glucose were dissolved in 200 cc. of water. Fifty cubic centimeters of this solution mixed with 5 cc. fuming HCl were heated according to the above-mentioned methods.

TABLE NO. II.—*Readings of an approximately 50 per cent solution of the various glucoses, before and after heating with acids. Temperature, 20° C.*

[Divisions on cane sugar scale.]

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.	No. 7.	No. 8.	No. 9.	No. 10.
Direct reading 200-mm. tube	85.7	85.5	80.1	86.55	85.3	84.4	90.4	84.9	86.15	42.00
Reading after inversion by first method	85.2	84.85	79.58	86	84.9	83.9	89.85	84.2	85.55	42.05
Difference	.5	.65	.52	.55	.4	.5	.55	.7	.6	......
Reading after inversion by second method	84.25	83.9	78.75	85.15	84	82.9	88.95	83	84.45	......
Difference	1.45	1.6	1.35	1.4	1.3	1.5	1.45	1.9	1.7	......
Reading after inversion by third method	83.75	83.2	78.1	84.60	83.5	82.15	88.35	82.25	83.75	......
Difference	1.95	2.3	2	1.95	1.8	2.25	2.05	2.65	2.4	......
Reading after boiling one hour with acetic acid (com.)	85.3	84.9	79.75	86.15	84.85	84.05	89.9	84.3	85.65	......
Difference	.4	.6	.35	.4	.45	.35	.5	.6	.5	......

The flasks were carefully calibrated and the duplicates agreed within the error of reading. The results are given in the above table. The solutions were of such a strength that the amount used was the same as would be used in the analysis of a substance containing approximately 50 per cent glucose.

It will be noticed that in every case, with the exception of No. 10, which contains no dextrin, the reading is sensibly diminished, introducing thereby an error in the estimation of sucrose in the presence of commercial glucose, varying from 1 to over 2 per cent, according to the amounts of the two present and the method of inversion used. While this may not be large enough to cause one to draw unjust conclusions from the analysis of commercial saccharin products, it is nevertheless desirable to eliminate it if possible, or at least to reduce it to a minimum.

Experiments were made to find out the effects of other acids in the hope of finding one that would bring about the inversion without affecting the reading of the glucose. The result showed, however, that even an acid which would not bring about complete inversion would sensibly diminish the reading of the glucose. Thus acetic acid will cause a diminution of reading almost as large as that caused by hydrochloric acid, although it does not seem to cause complete inversion. In no case was a smaller error introduced than by the first method above used.

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[1] Allen's Commercial Organic Analysis.

[2] Jour. des Fabr. de Sucre, XXXII, 1891, No. 35.

Since the error could not be eliminated by inversion with acids[1] an attempt was made to allow a correction for it. It is evident from the above results that the error is not a widely diverging one, if the samples can be regarded as average ones, and it could be largely diminished by allowing for the mean deviation.

It was necessary, however, before attempting this to find out what the effect would be when the amount of glucose present was varied. Solutions were made containing, approximately, 100, 50, 25, 10, and 5 per cent of glucose, respectively, and subjected to the action of acids as before. The average results expressed as divisions on cane sugar scale, were as follows:

	100 per cent.	50 per cent.	25 per cent.	10 per cent.	5 per cent.
Before ..	170.7	85	42.35	16.8	8.6
After...	169.4	84.45	42.15	16.78	8.65
	1.3	.55	.2	.02	....

The results with the various glucoses are sufficiently uniform to warrant one to introduce corrections which, while not eliminating the error, would reduce it to a minimum.

This correction may be easily calculated as follows:

It can readily be seen that the reading due to glucose present may be approximately determined by subtracting from the direct reading three-fourths of the difference between (or sum of) the direct and invert readings (supposing that the temperature be about 20°).

Let R equal the reading due to glucose, then the following formula deduced from a graphic representation of the above results will be found to give the correction (x):

$$x = \frac{R - 15}{130}$$

This correction must of course be subtracted from the numerator ($a \pm b$) in Clerget's formulæ. In order to test the accuracy of the above correction a number of mixtures of cane sugar and glucose were analyzed. Good results were obtained in every instance. The following expressed as divisions on cane sugar scale, will serve as examples:

Glucose.	Sucrose.	Direct reading.	Invert reading.	Approximate glucose reading.	Correction.	Per cent of sucrose found—	
						Without correction.	With correction.
Per cent.	*Per cent.*						
80	20	154.2	126.3	133	.9	20.82	20.18
50	50	138.4	70.75	87	.55	50.48	50.11
30	70	120.3	26.23	50	.27	70.20	70.00
10	90	109.0	11.8	19	.03	90.15	90.13

The results in Table No. 11 show the necessity of chemists using the same method of inversion. Even in the estimation of pure sucrose it has been shown by a number of chemists that the reading varies sightly according to the method used. In the presence of commercial glucose, however, the variation becomes much greater. When the inversion is accomplished by heating at 68° for ten minutes, the difference in reading due to the hydrolysis of the dextrin averages almost three times as great as when the first method is used, while the German method increases this to almost four times. It is barely possible that some of the widely-varying results published by the reporter on sugar in the report of the Association of Official Agricultural Chemists may owe a part of their discrepancies to these facts.

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[1] This error would be eliminated by the inversion brought about by O'Sullivan & Thompson's invertase process (Journal of the Chemical Society of London, Vol. 59, page 46). The method is not used to any extent, however, because of the time required as well as the increased labor of analysis.

CONCLUSIONS.

Some of the conclusions reached may be summarized as follows:

(1) An error is introduced in the estimation of sucrose in the presence of commercial glucose, due to a slight hydrolysis of the dextrin present during the process of inversion of the sucrose by acids.

(2) This hydrolysis, and consequently the error, is reduced to a minimum when the inversion of the sucrose present is effected by heating for ten minutes, the temperature being gradually raised so as to reach 68° at the expiration of this time.

(3) The results with the different glucoses are sufficiently uniform to warrant the introduction of a correction for the mean error.

(4) In order that the results may be comparable, chemists ought to strictly adhere to one method of inversion.

PART II.—ON THE ACTION OF ACETIC AND HYDROCHLORIC ACIDS ON SUCROSE.

While carrying out the above experiments with glucose several facts were noted in reference to the action of acetic and hydrochloric acids on sucrose, which it is interesting to compare with the conclusions reached by Bornträger,[1] Jungfleisch and Grimbert,[2] and Ost.[3] Inasmuch as we were working with an entirely different object in view, the conclusions reached were in no way influenced by the conclusions reached by the above writers.

While making an examination of a large number of saccharin products, the writers were led to believe from certain results that complete inversion could be effected by means of acetic acid. This view was contrary to the general opinion of chemists, although as prominent ones as Jungfleisch and Grimbert[4] claim that acetic acid completely inverts sucrose at 100°, and that it is preferable to other acids, since its presence is without effect upon the levulose of invert sugar. This conclusion was reached from the fact that the specific rotatory power of pure levulose corresponded almost exactly with that of levulose calculated from invert sugar, the inversion being effected by acetic acid. Hydrochloric acid gives a higher invert reading; because, in some way, it acts upon the levulose and increases very perceptibly its specific rotatory power. Exceptions to this view are taken by Maumené,[5] who calls attention to the fact that the invert sugar prepared from sucrose by action of acids is not composed of equal parts of levulose and dextrose. Ost[3] not only denies that acetic acid will produce complete inversion, but also claims that it acts perceptibly upon invert sugar. Ross[6] states, as the result of a number of experiments, that it was found impossible to secure complete inversion even when 10 per cent glacial acid was used and the flask kept immersed in boiling water for forty-five minutes.

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[1] Boh. Zeit. Zucker, Ind., 1891, 187. Bull. Assoc. Chim., 1892, 559.

[2] Compt. rend., **107**, 390 and **108**, 144.

[3] Ber. Chem. Ges., **24**, 1636.

[4] Compt. rend., **107**, 390.

[5] Journal des Fabricants de Sucre. Abstract from March 27, 1889.

[6] Report of the Association of Official Agricultural Chemists, Bul. 35, Div. of Chem., Dept. Agr., p. 146.

Those who claim that acetic acid does not effect complete inversion base their belief upon the fact that invert sugar, prepared by the action of hydrochloric acid upon sucrose, gives a higher reading than the corresponding solution obtained by the action of acetic acid. It was thought that perhaps acetic acid might form some compound with the constituents of the invert sugar, which might affect the reading of the polariscope. Proceeding upon this assumption, attempts were made to decompose such a compound. In the course of these experiments, which were conducted at great length, certain facts were noted which have been systematized in the following:

As stated above, Jungfleisch and Grimbert in particular, have called attention to the fact that the specific rotatory power of invert sugar, prepared by the action of hydrochloric acid on sucrose, is higher than that of the artificially made invert sugar. If, however, as Maumené claims, the invert sugar so prepared is not composed of equal parts of dextrose and levulose, then it is possible that the different reading is due to the peculiar compounds formed. In fact, one would not expect the readings to be identical under such assumptions.

Accordingly, experiments were undertaken to find out, if possible, whether the hydrochloric acid used in inversion affected in any way by its presence the reading of the invert sugar so prepared. Attempts were made in two different directions.

1. Five hundred cubic centimeters of a normal solution of sucrose (polarizing at 99.8) was subjected to inversion in 100 cc. flasks by adding one-tenth the volume of concentrated hydrochloric acid (sp. gr. 1.19) and heating for ten minutes, regulating the heat so that the temperature, 68°, was reached at the end of that time. These solutions were all poured into a large flask and thoroughly mixed, so as to obtain a perfectly uniform solution. Exactly 55 cc. of this solution were transferred to a 100–110 cc. flask, the flask filled to the mark with water, the solution thoroughly mixed and polarized. The experiment was repeated with the addition of 5 cc. of the same acid used in the inversion, previous to filling to the mark with water; again repeated with the addition of 10 cc., 15 cc., and 20 cc., respectively. Since the same solutions, same flasks and polarizing tubes were used, and the temperature of the solutions did not differ more than 0.5°, corrections being introduced for this variation, the chances of error in manipulation were very slight. The following readings expressed in divisions of cane sugar scale were obtained for the various solutions (temperature=20°):

Invert sugar (containing 5 cc. of acid)	—33.00
Invert sugar + 5 cc. additional acid	--33.90
Invert sugar + 10 cc. additional acid	—34.65
Invert sugar + 15 cc. additional acid	—35.60
Invert sugar + 20 cc. additional acid	—36.40

The reading is seen to increase with the addition of the acid, thus agreeing with the conclusion of Gubbe.[1] Moreover, the deviations caused by the addition of the different amounts of acid are fairly constant, varying from 0.75 to 0.95, or an average of 0.85. It was hoped that sufficiently uniform results could be obtained to allow the graphic representation, so that an estimate could be made on the effect of the original 5 cc. of acid added to bring about inversion. Since, however, the addition of a second, third, fourth, and fifth 5 cc. of acid each increases the reading of the invert sugar approximately 0.85, then we may conclude, with some probability, that the original 5 cc. of acid which effected the inversion would increase the reading by that amount.

2. Attempts were also made to solve the problem by neutralizing the acid present in the invert solution by the action of sodium carbonate.

Bornträger[2] has called attention to the fact that the rotatory power of the neutralized solution is higher than that of the corresponding acid solutions. By the addition of sodium carbonate, however, not only is the acid neutralized, but a corresponding amount of sodium chlorid is thereby added. Readings were taken first to find out the effect of the addition of the sodium chlorid. Then, by comparing this with the variation caused by neutralization with sodium carbonate, the effect of the acid could be deduced.

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[1] Ber. Chem. Ges., **18**, 2207.

[2] Boh. Zeit. Zucker, Ind., 1891, 187.

Five hundred cubic centimeters of inverted sugar solution were prepared as described above. It was found that 3.3 grams of pure dry sodium carbonate were necessary to neutralize the acid (5 cc.) present in 55 cc. of this solution. The experiments were conducted as in the above. Fifty-five cubic centimeters of the solution were transferred to a (100–110) flask, neutralized with sodium carbonate, the flask filled to the mark, the solution mixed thoroughly and polarized. The duplicates agreed within the limit of error in reading. The average readings on cane sugar scale were as follows (temperature=20°):

	Triplicates.		
	1	2	3
Original solution	—33.1	—33.	—33.2
Original solution + 3.3 grams sodium carbonate (neutral)	—33.7	—33.7	—33.85
Original solution + 6.6 grams sodium carbonate	—36.2	—36.	—36.3

The last reading was taken in order to compare the effects of the addition of sodium carbonate before and after neutralization. It is thus seen that, while the reading of the normal solution is increased 6° by the addition of the necessary amount of carbonate to neutralize the acid, the subsequent addition of a like amount increases the reading 2.5°. Before ascribing this difference in reading to the effect of the acid, it is necessary to find whether or not the sodium chlorid formed by the addition of the sodium carbonate to hydrochloric acid affected the reading.

Experiments were conducted with the same solution used above. Fifty-five cubic centimeters of the solution were introduced into the 100–110 cc. flask, and the amount of sodium chlorid corresponding to 3.3 grams of sodium carbonate, or 3.65, added, the flask filled and the solution polarized as before. The average readings cane sugar scale, agreeing within 0.1°, are:

Original solution	—33.1	—33.15
Original solution + 3.65 grams sodium chlorid	—35.1	—35.1

Since, then, the effect of the sodium chlorid was to increase the reading 2° and the acid remaining unneutralized while the reading was only increased 0.6° by the addition of the same amount of sodium chlorid, the acid being neutralized at the same time, the difference 1.4° may presumably be attributed to the action of the acid present.

This result is higher than the one deduced from the direct addition of acid given above, but it is undoubtedly the more correct of the two. It would thus seem that the acid undoubtedly increases the rotatory power, the probable increase of reading being about 1.4° on the direct scale for the normal solution.

Similar experiments were next conducted in order to find out the effect of acetic acid. The strength of the acid used may be inferred from the fact that 5 cc. of it required for neutralization 3.05 grams of dry sodium carbonate. The normal solution of sucrose was heated at 100° with one-tenth volume of this acid for about one hour, experiments showing that continued heating had but little effect upon the reading. By this treatment a reading for the normal solution was obtained on an average of 2° lower than when hydrochloric acid was used as the agent of inversion. The same solution which gave a reading of —33.1° when hydrochloric acid was used gave a reading of —31.1 when treated with acetic acid under the above conditions.

Experiments performed to discover whether the subsequent addition of acetic acid would affect the reading gave the following results, cane sugar scale:

Original invert solution (temperature 20)	—31.1	—31.
Original invert solution + 10 cc. additional acetic acid	—30.85	—30.8

The second reading is the reading obtained from a solution exactly the same as the first, 10 cc. of acetic acid being introduced.

It thus appears that while hydrochloric acid increases the reading by its presence, acetic acid decreases it by a small but perceptible amount. This result agrees with Ost, who criticises the statement of Jungfleisch and Grimbert that acetic acid has no effect upon invert sugar. In order to test this further, the acid was neutralized by sodium carbonate and the difference of reading noted. This result, united with the effect due to the introduction of the sodium acetate formed, gave the means of finding the effect of the acid.

The experiments were conducted as in the above, using same solution, same flasks, and same polarizing tubes. The following are the results obtained, cane sugar scale:

Original solution (temperature 20°)	—31.2	—31.1
Original solution + 3.05 grams sodium carbonate (neutral)	—32.65	—32.6
Original solution + (3.05 × 2) grams sodium carbonate	—34.8	—34.8

A marked difference is noted here as compared with similar experiments with hydrochloric acid. The neutralization of the hydrochloric acid increased the reading only 0.6°, while the neutralization of the acetic acid increased the reading over twice the amount, or 1.5°. The subsequent effects of the sodium carbonate upon the two solutions agree very well, being an increase of 2.5° in the case of hydrochloric acid against an increase of 2.2° in the case of acetic acid. This difference of 0.3° in the reading is accounted for by the fact that 3.3 grams of sodium carbonate were added in the hydrochloric acid solution, while only 3.05 grams were added in the acetic acid solution. If correction is made for this difference, the increase in readings differ only by 0.1°.

It was necessary also to find out the effect of the sodium acetate formed by the neutralization of the acetic acid by sodium carbonate. The readings on the cane sugar scale were as follows:

Original solution (temperature 20°)	—31.1	—31.1
Original solution + 4.72 grams sodium acetate	—32.4	—32.4

The sodium acetate used was the pure anhydrous salt.

It is thus seen that while the sodium carbonate increased the reading 1.5°, the introduction into the original solution of the same amount of sodium acetate as was formed increased the reading but 1.3°. The difference of 0.2° must be due to the presence of the acid.

The conclusion that the acetic acid by its presence lowers slightly the reading is confirmed by the fact that by the use of a weaker acid a maximum reading of —31.3° was obtained.

As might be expected the introduction of hydrochloric acid into a solution inverted by acetic acid increases the reading in a corresponding degree.

It is thus shown that the effect of hydrochloric acid upon a solution of invert sugar is to increase the reading, while acetic acid had an opposite effect. Moreover, the experiments show that the probable increase in reading due to hydrochloric acid under the conditions mentioned is 1.4°, while the probable decrease in reading due to acetic acid is 0.2°. In other words, if these two acids produced the same effects upon being heated with sucrose, there would still be a difference of reading of 1.6° due to the presence of the acid. This agrees fairly well with the actual difference observed.

In the light of these experiments it may be of interest to notice the question so often discussed, "Does acetic acid effect complete inversion?" If we suppose that no acid effects complete inversion unless it gives a reading such as that obtained by using hydrochloric acid, then most certainly acetic acid does not invert completely. If, on the other hand, we adopt as the reading of the invert solution the reading of the acid solution, corrected for the effect produced by the presence of the acid, then acetic acid gives more nearly the correct invert reading, and it is correct to affirm that acetic acid does invert sucrose completely.

Finally, the above results indicated that when Fehling's solution is used in the place of the polariscope, it would be a matter of indifference whether acetic or hydrochloric acid was used to effect the inversion of the sucrose. Experiments showed this to be true. Two solutions containing the same amount of sucrose were inverted with hydrochloric and acetic acids respectively. After inversion exactly 11.7 cc. of each solution were required to reduce 10 cc. of Fehling's solution. Moreover, when Fehling's solution is used for the determination of sucrose in presence of dextrin or commercial glucose, acetic acid is preferable to hydrochloric acid as the agent of inversion, since the process requires no attention, and the error introduced by the hydrolysis of dextrin is less even when the most favorable method of inversion is used, viz, heating for ten minutes, regulating the temperature so as to reach 68° at the expiration of that time.

Mr. HUSTON. There is one thing in this report which I think is of a good deal of importance, both in methods for sugar and methods for other material, and that is the extension which Mr. Spencer makes of the time after which tests will be made to judge as to whether the material has been brought to constant weight. I think we are too apt to make that period too short. We frequently weigh every hour, and if two of these weighings show no loss, we say the thing is constant weight. If you dried for four hours continuously instead of four hours in one-hour periods, you might find a very different condition of affairs. This is particularly true with regard to cattle foods. Mr. Spencer has done well to call our attention to these longer periods. It is the most important factor in our knowledge of whether we have the material at constant weight, particularly where the air bath is not very well ventilated, and where you have a considerable substance which offers only a small portion of its surface to the hot air.

No further discussion arising, the president called for the report of the reporter on nitrogen.

Mr. Bartlett then read his report:

REPORT ON NITROGEN

By J. M. Bartlett.

After being notified of my appointment as reporter on nitrogen for the year I carefully examined the records of the last meeting of the association for instructions on this division of the work, but a mere suggestion, from the reporter of last year on the Devarda method for determining nitric nitrogen was all I could find. This very naturally led me to believe that the association was pretty well satisfied with its methods for determining nitrogen, and this belief is still further strengthened when we consider what excellent results have been attained in the last few years in perfecting and improving the methods for determining this element. The results reported last year were, in the main, so satisfactory that it seemed to me at first unnecessary to send out more samples simply for analysis, and for a time I was strongly inclined to ask the members, who had any time to devote to the subject, to exert their energies in the direction of establishing a method for the determination of available nitrogen in mixed fertilizers—a subject which very much needs attention—as every chemist who has anything to do with fertilizer inspection is already aware. But, owing to other duties, I was unable to give this matter any attention until very late in the season, thereby making the time that could be devoted to such an investigation before the next meeting of the association decidedly too limited.

Calls for samples from chemists, who had time to devote to the subject and wished to compare results, began to come in, and on again looking over the work reported last year to see what most needed to be done, I observed that all the results on the samples free from nitrates were as close as anyone could ask, while those on the samples containing nitrates, although for the most part agreeing very well, in a few instances were a little wide; and I had also noticed that in my own work, when nitrates were present my results were liable to be a little further apart than when the substance was free from them. It seemed also desirable that methods for determining nitric nitrogen be again investigated, as such poor results were reported last year.

Accordingly, material was prepared for the purpose by making a bone superphosphate, drying and grinding, so it would pass through a millimeter sieve; then the organic nitrogen was carefully determined in this and a known amount of pure potassium nitrate added, so that the total nitrogen could be calculated in the mixture. The potassium nitrate was finely ground and dried, then evenly sifted over the bone superphosphate, spread on a rubber cloth, and mixed two hours by usual methods of rolling the cloth and use of a spatula. The bottles were then filled, the remaining portions removed from the cloth, the contents of the bottles again emptied on the cloth, mixed for one hour more, returned to the bottles and sealed. One of these samples was sent to nearly every station, and to other parties requesting them, together with a circular letter requesting the following determinations:

TOTAL NITROGEN.

First, by the Official method given in Bulletin No. 38, page 175 (U. S. Dept. Agr., Div. of Chem.), using 2 grams salicylic acid, 2 grams zinc dust, and 0.7 gram mercuric oxid or its equivalent in metallic mercury.

Second, by using 1 gram salicylic acid, 5 grams thiosulphate of sodium, and 0.7 gram mercuric oxid or its equivalent in metallic mercury.

Third, by the Gunning method modified for nitrates. The chemists taking part in this work were requested to be especially careful that their standard solutions be correctly standardized, and that the substance be thoroughly mixed with the sulphuric-salicylic acid before heat was applied or other reagents added. Determine nitric nitrogen, first, by Ulsch method modified by Street (Bulletin No. 35, p. 89, U. S. Dept. Agr., Div. of Chem.); second, by the Devarda method (Exp. Sta. Record,

p. 676) modified to use zinc dust and reduced iron instead of the Devarda alloy. In response to this request eighteen reports were received, and the results are given in tabular form below:

	Analyst.	Modified Kjeldahl zinc dust.	Modified Kjeldahl $Na_2S_2O_3$.	Modified Gunning.
		Per ct.	*Per ct.*	*Per ct.*
U. S. Department of Agriculture, Division of Chemistry.	T. C. Trescott	4.57	4.58	4.59
West Virginia Experiment Station	R. de Roode	4.66	4.77	4.64
Maryland State College	H. C. Sherman	4.77	4.67	4.70
Colorado Experiment Station	Wm. P. Headden	4.71	4.67	4.70
Virginia Experiment Station	Davidson & Watson	4.58	4.63	4.57
Wisconsin Experiment Station	F. W. Woll	4.73	4.67	4.60
West Virginia Experiment Station	T. F. Watson	4.68	4.66	4.64
North Carolina Experiment Station	F. B. Carpenter	4.76	4.77	[1]4.71
Michigan Experiment Station	W. L. Rossman	4.87	4.81	4.73
Cornell Experiment Station	G. W. Cavenaugh	4.69	4.71	....
New Jersey Experiment Station	E. B. Voorhees	4.76	4.78	4.74
Do	J. P. Street	4.78	4.77	4.74
Illinois Experiment Station	W. R. Powers	4.63	[2]4.46	[2]4.47
McCandless Laboratory, Atlanta, Ga	J. M. McCandless	4.60	....	....
Do	A. M. Lloyd	4.62	....	....
Kentucky Experiment Station	H. E. Curtis	4.66	4.68	4.65
Maine Experiment Station	L. H. Merrill	4.70	4.66	4.75
Do	S. H. T. Hayes	4.69	4.70	4.67
Do	J. M. Bartlett	4.70	4.71	4.67
Averages	....	4.69	4.70	4.67
Calculated per cent	....	4.71	....	....

[1] Ruffle method.

[2] Omitted from average; $Na_2S_2O_3$ added before acids.

Results of 15 analysts with zinc dust vary from calculated per cent less than 0.10
Results of 4 analysts with zinc dust vary from calculated per cent less than .... .16
Results of 15 analysts with thiosulphate vary from calculated per cent less than .... .10
Results of 1 analyst with thiosulphate vary from calculated per cent less than ...... .13
Results of 1 analyst with thiosulphate vary from calculated per cent less than .... .25
Results of 12 analysts with Gunning vary from calculated per cent less than .... .10
Results of 1 analyst with Gunning vary from calculated per cent less than .... .12
Results of 1 analyst with Gunning vary from calculated per cent less than .... .24

REMARKS OF ANALYSTS.

Dr. de Roode reports quite a large amount of work on the reporter's sample and a number of analyses by each method, by which he compares the use of metallic mercury and the oxid, also the regular method of digesting about 1 gram and distilling the whole, with that of weighing out a larger amount, 3 to 5 grams and distilling an aliquot part. His results agree very closely. He says: "I prefer this latter method when nitrates are present, as it enables me to take a larger quantity of the substance; therefore I get a better sample and somewhat diminish the errors of imperfect mixing. I am frequently troubled by getting a dark-colored residue with zinc dust that does not dissolve or become lighter colored as the digestion continues, and, in such cases, the results are liable to be low. I prefer thiosulphate, as it is cleaner and gives more uniform results than zinc dust. In making determinations by the Gunning method when the potassium sulphate and thiosulphate were added before the substance, the foaming was very bad and I think probably some loss of nitrogen. I prefer to add the potassium sulphate later in the process."

Dr. McDonnell writes: "We get a little more nitrogen by the modified Kjeldahl than by the Gunning method." In addition to the work on the reporter's sample the results of analyses of eleven mixed fertilizers containing nitrates, made in duplicate, comparing the modified Kjeldahl with the modified Gunning method, are given:

	Per cent.
Average by modified Kjeldahl method	3.07
Average by modified Gunning method	2.98

In working the modified Gunning method the thiosulphate and potassium sulphate were introduced into the flask with the fertilizer.

F. W. Woll. "The modified Gunning method gives me slightly lower results than the others. As regards time, the modified Kjeldahl method, using thiosulphate, is ahead."

Dr. Caldwell. "Our results are not very satisfactory, especially with the Gunning method, and I am somewhat puzzled to know where the trouble is."

FOREIGN CHEMISTS.

Only one report has been received from abroad, and that is from Dr. Fassbender, director of the Agricultural Experiment Station, Kempen a. Rhine, Germany.

METHOD EMPLOYED.

Place 1 gram of substance in a Kjeldahl flask, cover with 20 cc. of water, allow to stand a while and then add about 2 grams of coarsely powdered iron and 10 cc. dilute sulphuric acid. Warm gently until evolution of hydrogen ceases. Add 20 cc. strong sulphuric acid and a little paraffin to prevent foaming, and then proceed as by the regular method.

RESULTS.

No. 1, 4.66 per cent nitrogen.
No. 2, 4.72 per cent nitrogen.

DISCUSSION OF RESULTS.

Considered as a whole, I think the results are very satisfactory and as good as one could expect from so many different men in different parts of the country, working under varying conditions, and, as I have before said, I most always have found it more difficult to obtain closely agreeing results on mixed fertilizers containing nitrogen as nitrates than on those free from nitrogen in that form, due probably in part to the difficulty of getting the nitrates thoroughly and uniformly mixed with the other material. In the case of the material used in this work, I do not hesitate to say that when it was ready to go in the bottles it was as thoroughly and completely mixed as possible, but after bottling I observed that, owing to the bone being slightly coarser than the nitrate, there was a tendency to separation, and this fact may, in part, account for the disagreement of results reported by some analysts whose averages were very close to the actual amount. That good results could be obtained on the sample as sent out (when properly mixed before drawing the portion for analysis) was fully demonstrated by a large number of determinations made from many different bottles in our laboratory.

As will be seen by the table, the modified Gunning method gives a slightly lower average and, in a few instances, results that are too low to be called good. Also two chemists report that they could obtain no satisfactory results by this method. My opinion is, this trouble was caused largely by the official directions for the method given on page 175, Bulletin No. 38 (U. S. Dept. Agr., Div. of Chem.), which direct that the thiosulphate of sodium and potassium sulphate shall be put into the flask with the substance before adding the acids. Inasmuch as we heretofore, in any of the modified Kjehldahl methods for use on nitrates, have been especially cautioned to thoroughly mix the substance with the acids and allow to stand for a short time before adding the reducing agents, the directions seem to me truly remarkable and contrary to the whole theory on which the method is based. If I understand it correctly, the salicylic acid is added that it may unite with and prevent the escape of nitrogen oxids, freed by the strong sulphuric acid coming in contact with the nitrate, and hold them until they can be acted on and converted into ammonia by the reducing agents that are added later in the process.

It would seem, then, absolutely necessary that the nitrate be thoroughly mixed with and dissolved in the acids before a powerful reducing agent like thiosulphate is added that would be likely to decompose the salicylic acid and prevent its combining with the nitrogen oxids. I am aware that some chemists have obtained quite closely agreeing results by the method as given in the proceedings, but I think, in most cases, they worked with material containing a very small amount of nitric nitrogen, and probably quite closely agreeing results could have been obtained with no salicylic acid present.

Mr. Winton, in his work on the modified Gunning method (p. 173, Conn. Exp. Sta., Rep. 1893), says that he found it necessary in some instances to treat the substance two hours in order to dissolve all the nitrate before adding the reducing agents. In the work I have done I have usually found that if the nitrates were finely ground and thoroughly mixed with the acids they would, with frequent shaking, dissolve in about ten minutes.

To test the effect of adding thiosulphate before the acids are added, I took one of the sealed samples, such as were sent out for nitrogen determinations, pulverized and thoroughly mixed it, so as to remove any possible chance of variation from lack of uniformity in the sample. The results obtained are given in the table below, together with one by Mr. Merrill on another sample, and several by Mr. W. R. Powers, who writes he thinks his results are low from loss of nitrogen, as he added thiosulphate before the acids, but had no more material to test the matter:

Material.	Analyst.	Substance mixed with acid and allowed to stand ten minutes before adding $Na_2S_2O_3$ and K_2SO_4.	$Na_2S_2O_3$ and K_2SO_4 added with substance before acid.
		Per cent.	*Per cent.*
Reporter's sample	J. M. Bartlett	4.71	4.54
Do	do	4.71	4.48
Do	do	4.68	4.51
Do	do	4.68	3.60
Do	do	4.71	3.60
Do	do	4.73	4.06
Do	do		4.51
Do	do		4.65
Do	do		4.24
Do	do		[1]4.01
Do	do		[1]4.24
Do	do		[1]4.01
Do	do		[1]4.29
Potass-nitrate	do	13.77	[1]10.17
Do	do	13.78	[1]10.87
Reporter's sample	L. H. Merrill	4.75	3.99
Do	W. R. Powers	[2]4.63	[1]4.38
Do	do		[1]4.48
Do	do		[1]4.43
Do	do		[1]4.53
Do	do		4.43
Do	do		4.48
Do	do		4.38
Do	do		4.50
Do	do		4.48

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[1] Hg used instead of potassium sulphate.

[2] Modified Kjehldahl zinc dust.

NITRIC NITROGEN.

Several of the analysts reported determinations of the nitrogen present as nitrate, and the results, as will be seen by the table, were anything but satisfactory. The Ulsch method, modified by Street, was given another trial, and, as suggested by the reporter last year, the Devarda method modified, substituting reduced iron and powdered zinc for the Devarda alloy, was used. The following results were obtained:

	Ulsch-Street.	Devarda Fe and Zn.	Devarda.
	Per cent.	*Per cent.*	*Per cent.*
T. C. Trescott	1.98	2.02	2.05
R. de Roode	1.95	....	....
Wm. P. Headden	2.11	2.14	....
Davidson & Watson	1.06	1.97	....
T. F. Watson	1.91	....	....
J. P. Street	1.95	....	....
Do	1.90	....	....
Do	1.93	....	....
W. R. Powers	1.40	2.08	....
J. M. McCandless	1.66	1.64	....
A. M. Lloyd	1.63	1.64	....
Calculated	1.91	....	....

After receiving the above unsatisfactory results I made a few experiments to learn, if possible, where the trouble was. As will be seen by the table my first results by the Ulsch-Street method were too low, and the loss was due, I think, to incomplete distillation. I found it necessary to distill a very long time, and nearly to dryness, to get all the ammonia over. I had better success using sulphuric acid, 1 part acid to 2 of water, instead of 1 to 1, as Mr. Street recommends. I added the same amount of magnesium oxid as when stronger acid was used. With the Devarda method modified, using 2 grams zinc dust and 2 grams reduced iron, I obtained very uniform results, but in every case too high, probably due to the organic nitrogen compounds in solution being converted into ammonia by boiling with the sodium hydrate. The nitrates were leached out in every case in working with this method. Notwithstanding the fact the Devarda method modified gives results a little higher than the calculated per cent, I think it safer than the Ulsch-Street, as the latter seems to be liable to give results much too low even in the hands of men who have had considerable experience with it. With the carefully mixed sample I obtained the following:

Ulsch-Street.	Devarda modified.
Per cent.	*Per cent.*
1.66	2.10
1.68	2.10
1.82	2.05
1.82	2.01
1.82	....
1.96	....
1.94	....
1.98	....
Calculated per cent: 1.91	....

Mr. S. H. T. Hayes has also made a few experiments by Ulsch–Street method, and he obtained the following results:

	Per cent.
By the method as given by Mr. Street	1.98
Using a more dilute acid one-half	1.98
By using 2 grams of iron	1.91 1.95
By using 10 cc. strong acid	1.92 1.94
By regular method not boiled with acid	1.99
According to directions, except boiled one-half hour with acid.	1.99 2.00

Mr. Hayes says: "I found it necessary in every case to distill nearly to dryness; otherwise the results were low."

I think as yet we have no Official method for determining nitric nitrogen, and, as we are frequently called upon in the fertilizer work to make this determination, it seems to me it would be well for the association, if not satisfied with any of the methods given, to investigate the matter a little more fully and recommend one to the next meeting.

RECOMMENDATIONS.

As no work has been done on the method for determining nitrogen in the absence of nitrates, I have no changes to suggest.

(1) I would recommend that the quantity 0.7 to 3.5 of material taken for analysis by the Kjeldahl method and its modification be made uniform.

(2) On page 173 (Bulletin No. 38, U. S. Dept. Agr., Div. of Chem.) under (2) change standard ammonia to standard alkali.

(3) Page 174 (same bulletin, under paragraph 1), third line, change 22 to 20.

(4) On page 175 (same bulletin) in the directions for the Kjeldahl method modified to include the nitrogen of nitrates, I would suggest that the words "until thoroughly mixed with the acid" be inserted after the word "shake" in the third line in place of the word "thoroughly."

(5) Change the comma after the word "thiosulphate" in the same line to a semicolon, and the semicolons before "then" and after "acid" in the next line to commas.

(6) In the Gunning method modified to include the nitrogen of nitrates I would recommend to be changed to read as follows: "In a digestion flask holding from 250 to 500 cc. place from 0.7 to 3.5 grams of the substance to be analyzed according to the amount of nitrogen present. Add 30 to 35 cc. of salicylic acid mixture, namely, 30 cc. sulphuric acid to 1 gram of salicylic acid, shake until thoroughly mixed and allow to stand five to ten minutes with frequent shaking; then add 5 grams thiosulphate of sodium and 10 grams of potassium sulphate. Heat very gently until frothing ceases, then heat strongly until nearly colorless. Dilute, neutralize, and distill the same as in the Gunning method."

As the method presented by Dr. Fassbender seems to be exceedingly simple, it would perhaps be well for the association to give it a trial next year.

Mr. Huston. I assume that the committee will act upon the recommendations upon having their attention called to them.

The Secretary. All recommendations are referred to the committee without motion, on account of last year's resolution requiring it.

The president called for any other papers on nitrogen.

Mr. Bartlett presented a paper sent by Mr. F. S. Shiver, of Clemson College, South Carolina.

THE DETERMINATION OF NITRIC NITROGEN BY THE ZINC-IRON METHOD.

By F. S. Shiver.

The reporter for 1893 on nitrogen for the Association of Official Agricultural Chemists voiced, I think, the sentiment of the association when he expressed the opinion that if a rapid, simple, and accurate method could be found for the determination of nitric nitrogen in commercial nitrates and fertilizers it would be a valuable addition to the present methods of the association. It is with the application of the Zinc-iron method to the former (nitrates) that this article chiefly deals, as I have not had sufficient time at my disposal to investigate thoroughly its application to the latter (mixed fertilizers), though I see but one reason for its possible failure in this connection, and that is the liberation of ammonia by the action of the caustic soda upon the organic nitrogenous materials that may have entered the aqueous solution in the course of leaching out the nitrate. This plus error in some cases, as I shall show, would amount to practically nothing, but would increase proportionately as the amount of soluble organic nitrogenous material in the fertilizer under examination. However, from the few results I can gather I am inclined to look upon it favorably in this connection also.

The principle of the Zinc-iron method and the many modifications that have been proposed has long been known and made use of in many different ways, and is based upon the fact that when a nitrate is heated with a strong alkaline solution and zinc added, ammonia is evolved. It has been shown, however, that by the use of zinc alone the quantity of ammonia evolved is not a constant measure of the nitric acid present. It appears that Vernon Harcourt and Siewert, working independently, arrived at the conclusion that by the use of zinc and iron the reaction was perfect (J. C. S., 1862, 381, An. Chem. u. Phar., cxxv, 293). The modification I have made use of in this work, and to which no claim of originality is made, is the following:

Five grams of the nitrate are dissolved in water and made up to 500 cc., 50 cc. are transferred to a Kjeldahl distilling flask, 5 grams each of zinc and iron added, the former in the shape of dust (zinc dust), the latter as fine iron filings, 75 cc. of water and 80 cc. NaOH of about 1.3 sp. gr. then added, and the flask is now connected with a condenser, to which is attached an Erlenmeyer flask containing standard acid (20 cc. of sulphuric acid containing 0.01999 gram SO_3 to the cubic centimeter was used in this work), and after thorough mixing is allowed to stand one hour in the cold; at the expiration of this time a gentle heat is applied, and after maintaining this for awhile the contents of the flask are brought to a boil and the boiling continued until 100–125 cc. of distillate are obtained. I have found it necessary to observe this precaution, else there is danger of soda being carried over mechanically, due to the violent reaction which takes place when heat is first applied. The only other precaution I have found necessary to insure accuracy of results is the use of a bulb tube, with two bulbs with a loose plug of asbestos in the lower bulb with which to connect the distilling flask to the block-tin pipe of the condenser. I have found from several blank experiments that by proceeding in this way there is no danger of soda being carried over mechanically; it is necessary, however, to make a few blank experiments to ascertain the amount of nitrogen in the reagents used, and these should be conducted under the same conditions as all subsequent work. In this work I have found a uniform blank of 0.0005 gram nitrogen, which has been used to correct all the results obtained in the course of this work. With proper attention a determination can be made by this method in from two to three hours, including weighing of sample.

Chlorids and sulphates do not interfere with the accuracy of the process. As is no doubt well known these two sources of impurity in commercial nitrates interfere very seriously with the results obtained by an equally simple and accurate method, namely, the silica fusion method; here, unless the most scrupulous attention is paid to the ignition, the results will be too high, owing to the loss of hydrochloric acid in addition to the expected loss of the nitric acid of the nitrate. It has also been suggested by some, though I have not seen any figures on the subject, that chlorids interfere with the accuracy of the modified Kjeldahl in the case of high percentages of nitrogen; from the results obtained by myself and here appended I do not think such is the case.

KNO_3 c. p. recrystallized and thoroughly dried at 130° C. was used in these experiments.

0.3 gram KCl added	13.84 per cent N.
0.3 gram KCl added	13.84 per cent N.
0.3 gram NaCl added	13.81 per cent N.
Theory	13.88 per cent N.

The same sample of KNO_3 was used in the following experiments:

Five grams of the carefully prepared salt was then taken and dissolved in 500 cc. water and 50 cc. taken for each determination by the Zinc-iron method; in the case of the modified Kjeldahl portions of 0.5 gram was weighed out and digested as usual. Following are the results:

	Per cent N.
Theory	13.88
Found	13.86
Found	13.83
Found	13.81
Found	13.84
Found	13.84
Found	13.81
Found	13.88
Found	13.88
Average	13.84

The greatest variation in these eight results is 0.07 per cent below theory in two cases; in two cases the results are identical with theory; in one case 0.02 per cent below; in one case 0.05 per cent below; in two cases 0.04 per cent below. Average of all results is 0.04 per cent below theory. Another sample of KNO_3 c. p., was recrystallized twice, and after thorough drying at 130° C. was subjected to analysis, proceeding in the manner as described above with the first sample.

Following are the results:

	Per cent N.
Theory	13.88
Found	13.83
Found	13.86
Found	13.83
Found	13.83
Found	13.83
Found	13.83
Average	13.84

Five of these results are 0.05 per cent below theory and one 0.02 per cent below; average of all, 0.04 per cent below.

Therefore, comparing the two series of results, we have the following conclusions to draw: Six results are 0.05 per cent below theory; two 0.02 per cent below, two 0.04 per cent below, two 0.07 per cent below, and the remaining two identical with theory. Average of all results is 0.04 per cent below theory.

In order to ascertain as carefully as possible the exact nitrogen content of these samples, the Kjeldahl modified for nitrates was used, as it is easy of manipulation and accurate in its results. The first sample of potassium nitrate was used, and the following are the results:

Zinc was used as reducing agent in column marked zinc; $Na_2S_2O_3$ was used in column marked $Na_2S_2O_3$.

	Theory, 13.88 per cent N.	
	Zinc used.	$Na_2S_2O_3$ used.
	Per cent N.	*Per cent N.*
Found	13.81	13.81
Found	13.81	13.88
Found	13.84	13.81
Found	13.84	....
Average	13.83	13.83

It will be seen that four results are 0.07 per cent below theory, two 0.04 per cent below, and one identical with theory. The average of results by using zinc is the same as was obtained by using $Na_2S_2O_3$, 13.83 per cent N in both cases; 0.05 per cent N below theory. To compare the two methods we have the following results:

	Per cent N
Average of modified Kjeldahl	13.83
Average of zinc-iron	13.84

The results by the two methods, therefore, agree very closely, differing only by 0.01 per cent N, and falling 0.05 per cent below theory in case of modified Kjeldahl, and 0.04 per cent in case of the Zinc-iron method. The next thing that was done was to submit a few samples of commercial sodium nitrate to analysis, comparing the Zinc-iron method with the modified Kjeldahl method. Following are the results:

	Per cent N.	Per cent N.
First sample:		
Zinc-iron method	16.08. and	16.05
Mean		*16.07*
Modified Kjeldahl method	15.98 and	15.96
Mean		*15.97*
Second sample:		
Zinc-iron method	15.92 and	15.89
Mean		*15.91*
Modified Kjeldahl method	15.87 and	15.92
Mean		*15.90*
Third sample:		
Zinc-iron method	15.89 and	15.85
Mean		*15.87*
Modified Kjeldahl	15.80 and	15.76
Mean		*15.78*

In the first sample difference in results between the two methods was 0.10 per cent N, in the second sample 0.01 per cent N, and in the third sample 0.09 per cent, the Zinc-iron method giving the higher result in each case. Average of all results by Zinc-iron method on commercial $NaNO_3$, 15.95 per cent N; by modified Kjeldahl, 15.88 per cent N; difference in average results is 0.07 per cent N. Considering the slight errors involved in working on one-half gram of material and in the use of any titration process and the subsequent doubling of the result where one-half gram is used, it seems to me that this agreement is very satisfactory.

A sample of fertilizer consisting of acid phosphate and nitrate of soda without any organic nitrogenous material was then taken and analyzed by the Dumas or absolute method and by the Zinc-iron method. The following was the method of procedure adopted for the Zinc-iron method: 10 grams of the fertilizer are leached with water until 500 cc. of leachings are obtained, and after having satisfied yourself of the absence of nitrates by the sulphuric acid and ferrous sulphate test, 25 or 50 cc., according to the amount of nitrate in the sample, is taken and placed in a Kjeldahl distilling flask and treated in the same way as described in the first of this article. Following are the results:

	Per cent. N.
Absolute method	7.69
Zinc-iron method	7.63

Difference in results by the two methods is 0.06 per cent. N.

I would also add here the result obtained by Mr. Jones on the sample sent out by last year's reporter on nitrogen for testing this point of nitric-nitrogen estimation in fertilizers. Mr. Jones obtained 3.44 per cent N by this method; theoretical amount was 3.46 per cent N (Bulletin No. 38, U. S. Dept. Agr., pp. 37 and 38). I hope in the future to supplement the few results here obtained on complete fertilizers with a larger number, and in consequence be in a position to judge more thoroughly of its accurate application to the determination of nitric nitrogen in complete fertilizers.

From the results here reported and those obtained elsewhere it would seem as if the accuracy of its application to the determination of nitric nitrogen in nitrates has been established, and though the results are few in regard to complete fertilizers, yet the method seems promising and is deserving of a more extended study than I have been able to give it. The easy adaptability of the Kjeldahl apparatus to this method is something of an argument in its favor, in that by its adoption by the association no new outlay of apparatus and consequent expenditure of money would be necessary.

In conclusion I would express the desire that the few results nere reported may be the means of giving this method a more thorough investigation at the hands of the members of the association.

Mr. Ross gave an abstract of a paper summarizing methods of standardizing acid and alkali solutions, written by his assistant, Mr. H. K. Miller:

THE STANDARDIZATION OF NORMAL ACID AND ALKALI SOLUTIONS.

By H. K. Miller.

The experiments outlined below were conducted for the purpose of practically testing the relative merits of some of the leading processes employed in determining the strength of the standard acid and alkali solutions used in nitrogen estimations.

The acid employed in this work of investigation was deci-normal hydrochloric acid, prepared in the manner prescribed in the official methods of the Association of Official Agricultural Chemists.

An amount of the acid was employed sufficiently large to give a precipitate of AgCl weighing not less than 0.7 gram, and the determinations were repeated until the results of several weighings showed an extreme variation of less than 1 milligram.

After several dilutions and redeterminations of chlorin, an acid was obtained containing 3.6452 grams of HCl per liter, as calculated from the weight AgCl in an aliquot of the acid. Tenth normal solutions of ammonia, potash, and soda were prepared in the usual manner with the exception that in the case of the soda solution the alkali was prepared by the action of metallic sodium upon water, and in the preservation of this solution carbon dioxid was excluded as far as possible.

In the first check determination of the strength of the standard acid sodium carbonate was used, the dry c. p. salt prepared by Trommsdorf being employed. A portion of this salt was heated at a low red heat and cooled in a desiccator; 5.3

grams was then weighed up, dissolved in water, and made up to 1,000 cc. On titrating with acid it was found that 50 cc. of n/10 HCl required 50.5 cc. of the sodium carbonate solution as compared with 50 cc., the theoretical amount. Cochineal was used as an indicator, but the reaction was not at all sharp nor well defined. Potassium tetroxalate was next used to check the strength of the standard alkali solutions, several samples of the salt having been very carefully prepared. In the preparation of this salt, c. p. potassium dioxalate and a somewhat larger weight of c. p. crystallized oxalic acid were used. The two substances were dissolved simultaneously in boiling water, and the solution was concentrated by evaporation on the water bath. The hot solution was then allowed to cool very slowly. The mother liquor and the greater proportion of the elongated crystals were now removed, and the remaining crystals were dissolved in water. Evaporation and crystallization were now proceeded with as before and the recrystallization was continued until the third successive operation had been completed. The crystals were now washed with water and dried by means of filter or blotting paper, and also over sulphuric acid. Five-tenths gram of a sample of this material was dissolved in water and titrated with n/10 soda solution, using phenolphthalein as an indicator.

Seven separately conducted weighings and titrations gave a mean of 59.03 cc. sodium hydrate solution as against a theoretical equivalent of 59 cc.

Several separate experiments with another sample of potassium tetroxalate, obtained by a separate preparation, gave us a mean result 29.6 cc. of n/10 soda solution required as compared with 29.51 cc., the amount required by theory for 0.25 gram of the tetroxalate.

Another series of experiments with the potassium tetroxalate, titrating with caustic potash solution, showed a like close agreement between the amounts of alkali actually and theoretically required.

A series of experiments was next conducted in which a number of separate portions of ammonium ferrous sulphate were weighed up, dissolved in water, and the ammonia distilled off after the addition of caustic alkali to the solutions. Five-tenths of a gram of the salt was used in these tests, and the amounts of standard n/10 acid neutralized by the ammonia distilled off showed variations of from 0.05 to 0.7 cc. when compared with the calculated amounts.

A number of experiments with ammonia-iron alum gave even less satisfactory results. The experiments in testing the strength of the standard solutions were made with the employment of the electrolytic method proposed by Hart and Croasdale. The copper sulphate employed was a c. p. article manufactured by Trommsdorf; this was dissolved in water and recrystallized twice.

The electrolysis of the copper sulphate solution was effected in the prescribed manner, the acid liquid together with the washings being titrated with standard alkali at the completion of the operation.

Five-tenths gram of copper sulphate was used in each experiment, the theoretical amount of n/10 normal alkali required to neutralize the acid formed being 40.07 cc. The mean of six separate tests was 40 cc., showing a variation of only a little more than half a tenth of a cubic centimeter of decinormal alkali. The reduced copper was also weighed as a check on the results secured by titration. It was found, however, that the deposit was somewhat dark in color and sometimes a little inclined toward sponginess, and when weighed under these conditions the results secured were invariably too high. If this film of copper, however, is dissolved in dilute nitric acid and the solution still further diluted, a very bright and pure deposit of metallic copper can be easily secured on the repetition of the electrolysis. The mean of four separate tests executed in this manner gave as the weight of metallic copper secured from 5 grams of the sulphate, 0.1275 gram. The average of five determinations was 0.12704 gram (included in this is a rather low result). The calculated weight is 0.12737 gram.

The results of these tests indicate that with a pure salt, and with the observance of proper precautions in the execution of the method, this process can be utilized to great advantage in testing the strength of standard solutions.

An amount of copper sulphate sufficient to yield theoretically one liter of n/20 acid was then subjected to electrolysis and the acid liquid made up to 1,000 cc. With such a large amount of copper sulphate the process did not yield as satisfactory results as with smaller quantities, 100 cc. of the acid corresponding to only 49.55 cc., of deci-normal alkali. Potassium bitartrate was the substance last employed in this series of experiments. The salt was prepared from c. p. potassium chlorid and tartaric acid, the precipitation being effected by alcohol in the presence of acetic acid. The precipitate is washed well with alcohol, both by decantation and on the filter, the mode of procedure being quite similar to that followed in the determination of tartaric acid in baking powders, as described in Bulletin 13, Chemical Division, U. S. Department of Agriculture. The well-washed salt is dried at 100° C. and several equal portions are weighed out and dissolved in hot water. The mean of three titrations with n/10 alkali gave 26.45 cc., as against the theoretical amount of 26.5 cc. A 1/40 normal solution and a 1/20 normal solution prepared from the potassium bitartrate and tetroxalate, respectively, showed no appreciable difference in strength from the standard acid diluted to a corresponding extent.

The PRESIDENT. Are there any further remarks or papers on this subject? If not, the matter of cattle foods is next in order.

Mr. HUSTON. Before leaving the matter of fertilizers I should like to call attention again to the preparation of the sample. We are limited to 1.4 grams as the quantity we are to take; I believe this is too small. If we take larger quantities of many goods they can be better sampled. It has seemed to me one of the best points in the Stassfurt method that it starts with 100 grams for an analytical sample. In connection with the matter of sampling comes the preparation of the sample. We have omitted all preparation of fertilizer samples except four lines on page 169. I find that over 20 per cent of my samples can not be prepared by this method. It is impossible to put them through a 1 mm. mesh sieve. I think we must allow ourselves a little latitude in the handling of such samples. We are governed in our laboratory by circumstances. We try to get a thoroughly good mixture if we have to use a one-fourth-inch sieve. We use the smallest sieve the stuff will go through without making it cake or stick. Most of it can be handled through a 14 mesh to the inch sieve without much trouble. Very often we can work that down through a No. 25 sieve, which is approximately equal to a 1 mm. mesh sieve with round hole. Many of our samples we handle through a No. 25 sieve as the nearest approach to 1 mm. mesh sieve. We ought to be allowed considerable latitude as to the amount of the material weighed out in the case of samples which can not be passed through a 1 mm. mesh sieve.

Mr. PAYNE. I will describe my process as a suggestion in the discussion. When I get such a sample I sift out the dryer portions and go over it in detail so that every particle of the sample is rubbed down. Then I have a homogenous mass a good deal like putty. Then I go over it again with a spatula, using the dryer material to make the damp stuffs become well distributed through it, and in that way I can get a finely granulated substance that works very well. Of course I dare not dry it at 100° and powder it.

Mr. VAN SLYKE moved that, in view of the crude condition in which many of the methods were, the president appoint a committee to go over the printed methods and arrange them in better shape, striking out such portions as are superfluous, and that this committee report next year.

Carried

The president called for the paper of the reporter on cattle foods. The reporter being absent on committee service, the paper of the reporter on fermented liquors was taken up, and, in the absence of Mr. Colby, the report was read by the secretary.

UNIVERSITY OF CALIFORNIA, COLLEGE OF AGRICULTURE,
AGRICULTURAL EXPERIMENT STATION,
Berkeley, July 30, 1894.

DEAR SIR: Herewith inclosed please find report on fermented liquors.

I would like to call attention to one or two points, but not to recommend any changes in the methods of analysis.

In determining the acidity of a wine, if decinormal alkali is used with litmus indicator the neutral point is easily ascertained by making successive tests of a portion (20 cc.) of the liquid in a small porcelain dish against one drop of decinormal alkali; the change of color for a white wine is to deep blue, for a red wine to green or bluish-green if the neutral point of the whole liquid has been reached.

The method I have used here for the glycerol determination, seems to give very close results when the work is duplicated. After adding lime or magnesia to the wine in the dish, add 10 grams coarse $CaCO_3$, evaporate and stir till dry lumps remain, remove all the substance into a Soxhlet's extraction tube; the last part adhering to the dish loosens easily with warm alcohol. After extraction with alcohol, cooling and allowing to stand with ether, filter and wash into weighed flask; evaporate on water bath gently heated (60° C.); a current of dry air is useful toward the end of the evaporation. Dry finally at 100° C. till 0.002 gram is the loss for two hours' heating; the weighed crude glycerol is washed with alcohol into a pint dish, incinerated, and ash subtracted from the crude glycerol. You, of course, will recognize most of this as coming from Allen's Com. Org. Anal.

Hoping the association meeting is well attended, I am, sir,

Very truly yours,

GEO. E. COLBY.

Prof. H. W. WILEY,
Washington, D. C.

REPORT ON FERMENTED LIQUORS.

By GEO. E. COLBY.

Owing to the delay caused by the resignation of Mr. C. A. Crampton, reporter upon this subject, it was decided to make the work as brief as possible. With this object in view, one analysis of a dry red wine (Blend of one-half Cabernet Sauvignon and one-half Crabb's black Burgundy, vintage of 1892) was undertaken. The grapes from which this wine was made were grown at Mission San Jose, Alameda County, Cal., and the wine made in the wine cellar of the Agricultural Experiment Station of the University of California.

To those requesting it a sample was sent, and with the wine the following letter:

UNIVERSITY OF CALIFORNIA, COLLEGE OF AGRICULTURE,
AGRICULTURAL EXPERIMENT STATION,
Berkeley, April 11, 1894.

DEAR SIR: I send you to-day by express a package containing a sample of dry red wine, marked "Red wine," vintage of 1892.

I should be pleased to have you make an analysis comprising the determinations named in my letter of March 22, 1894. The association methods given in Bulletin 38, pages 207–210, should be strictly followed for this work. For nitrogen determination use 100 grams of wine, evaporate to dryness on a water bath in Kjeldahl flask, after which operation proceed as usual. Please give nitrogen percentage and albuminoids equivalent to same. The dealcoholized wine, or residue left after the alcohol determination, serves, when made to original volume, for spindle determination of extract (body) and tannin. Please read, too, the specific gravity of the distillate and wine with the spindle.

Any remarks you desire to make upon the methods or results will be acceptable. Please send reports so as to reach me by July 1, 1894.

Very truly, yours,

GEO. E. COLBY,
Reporter on Fermented Liquors.

Eight out of fourteen to whom samples were sent reported. The following-named chemists, not connected with any agricultural experiment station, requested and received samples of this wine for analysis, and their results are given below in the tables: Messrs. H. G. Shaw, of the Toledo high school, Toledo, Ohio; W. C. Blasdale, of the chemical department, University of California, and Marvin Curtis (late assistant chemist in this station), wine chemist, San Francisco, Cal.

The table just below shows the results obtained by the various chemists who reported to me:

Analyses of red wine (after association methods).

Analyst.	Specific gravity.	Alcohol.		Tannin.	Acid.		Extract.	Ash.	Nitrogen.	Albuminoids equivalent to nitrogen.	Glycerol.	
		By weight.	By volume.		Total as tartaric.	Volatile as acetic.					Crude.	Less ash.
		P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
W. C. Blasdale, California	0.9970	8.16	10.15	0.184	0.664	0.070	2.54	......	0.035	0.219	1.07	1.03
F. T. Bioletti, California	.9975	8.03	10.10	.170	.650	.073	2.60	0.32	.037	.231	.92	.82
Geo. E. Colby, California	.9964	8.07	10.15	.186	.619	.069	2.77	.27	.0405	.250	.90	.85
Marvin Curtis, California	.9973	8.19	10.29	.191	.615	.086	2.71	.32	.041	.258	.92	.82
H. J. Patterson, Maryland	.9978	9.06	11.26	......	.795	[1].049	2.54	.30	.0385	.2406	......	......
H. H. Nicholson, Nebraska	.9975	7.88	9.80	......	.991	.241	2.65	.31	.041	.256	......	......
Messrs. Krug and Bigelow, U. S. Department of Agriculture	[2].99748	[2]8.26	[2]10.36	[3].188	[4].680	[3].064	[3]2.62	[3].31	[3].039	[3].2438	......	[3].649
H. G. Shaw, Toledo, Ohio	.9963	8.54	10.62	......	.706	.077	1.69	......	......	.348	......	......
Mr. Cavanaugh, Cornell University	.9968	7.99	9.93	.078	.759	.107	2.52	.313	.057	.3563	......	......

[1] Determined in the alcohol distillate. [2] Bigelow. [3] Krug. [4] Carman.

While it is to be regretted that more have not participated in this work, the results show, in the main, quite satisfactory agreements, and as they are not numerous or hard to compare they are given without making an average.

The percentages of total acidity and volatile acid in the sample sent to Nebraska are very high—the wine must have become sour either during transportation or in the laboratory.

The quantities of glycerol show a wide range, from 0.649 per cent (U. S. Dept. Agr.) to 1.03 per cent (Blasdale, Cal.), indicating, perhaps, that the method of its determination might well be further studied, or controlled.

Most of the results of the direct determination (by evaporation) of the extract are quite close, and, as is to be expected, in no case reach as high as the figures for solid contents in the wine as determined by the pyknometer and spindle (indirect methods) and which are reported herewith in the table of comparison of methods.

A few notes have accompanied some of the results; in reporting the acidity of this wine the Maryland chemist does so as follows: "Total acids 100 cc. equals 106 cc. decinormal NaHO. Volatile acids 100 cc. equals 8.3 cc. decinormal NaHO (determined in the alcohol distillate)." Mr. Krug, of the U. S. Department of Agriculture, makes this observation upon the indirect determination (by pyknometer or hydrometer) of extract: "Skalweit, Hannoversche Zeitschrift 'Wider die Nahrungsfälscher' recommends adopting a mean between Hager's and Schulze's tables for the indirect extract, and by so doing the figure obtained for this wine is very nearly the same as that obtained by the direct method." These methods give for the indirect extract "according to Hager 2.48 per cent, according to Schulze 2.92 per cent, averaged at 2.70 per cent" against 2.62 per cent direct extract.

That portion of the work bearing upon the methods of determining the specific gravity, alcohol and extract (body) has been only partially done, mostly for the reason that some of the chemists carrying on this investigation did not have a set of spindles, as recommended, for the purpose.

The table below shows what has been accomplished upon the points involved:

Comparative determinations of specific gravity, alcohol and extract (body) by pyknometer (association method) and by "spindle," or hydrometer.

	California.					
Analyst	F. T. Bioletti.		W. C. Blasdale.		George E. Colby.	
Method	Association.	Spindle.	Association.	Spindle.	Association.	Spindle.
WINE ANALYSIS.						
Specific gravity of wine	0.9975	0.9968	0.9970	0.9967	0.9964	0.9965
Specific gravity of dealcoholized wine	1.0117	1.0110	1.01085	1.0110	1.0113	1.0110
Specific gravity of distillate 15° C	.9865	.9867	.98645	.9863	.9864	.9864
Alcoholometer reading	....	10.10	....	....	....	10.14
Alcohol, weight per cent	8.03	8.00	8.16	8.24	8.07	8.07
Alcohol, volume per cent	10.10	9.95	10.15	10.25	10.15	10.15
Solid contents by spindle, per cent	....	2.90	....	2.86	....	2.82
Solid contents of dealcoholized wine (pyknometer), per cent	3.00	....	2.82	....	2.90	....

Comparative determinations of specific gravity, alcohol and extract (body) by pyknometer (association method) and by "spindle," or hydrometer—Continued.

	Maryland.		Nebraska.		U. S. Department of Agriculture.	Cornell University.	
Analyst	H. J. Patterson.		H. H. Nicholson.		W. D. Bigelow.	Mr Cavanaugh.	
Method	Association.	Spindle.	Association.	Spindle.	Association.	Association.	Spindle.
WINE ANALYSIS.							
Specific gravity of wine	0.9978	0.9975	0.9975	0.9975	0.99748	0.99675	0.9970
Specific gravity of dealcoholized wine	1.0111	1.0110	1.0111	1.0112	1.01129	....	....
Specific gravity of distillate 15° C	.9852	.9854	.9868	.9865	.98619	....	....
Alcoholometer reading	....	....	....	....	....	....	....
Alcohol, weight per cent	9.06	8.90	7.88	....	8.26	7.99	8.08
Alcohol, volume per cent	11.26	11.05	9.80	....	10.36	9.93	10.00
Solid contents by spindle, per cent	....	2.84	....	2.89	....	....	....
Solid contents of dealcoholized wine (pyknometer), per cent	2.87	....	2.83	....	(2.90)	....	....

In as far as the results go they are very satisfactory, the general uniformity being broken only by the alcohol, as reported by the Maryland Station, which gives a number, both by the association and spindle methods, about 1 per cent more than the others report. These results, meager as they are, tend to show that for the important determinations in question the spindle can be depended upon for accurate returns when observed at the correct temperature; besides, as is well known, its use saves much time. The ordinary spindle is not delicate enough for such work, and for those who desire to use the spindle method we would state that a very good set of hydrometers, such as we have used for a number of years in this laboratory, is described in a catalogue by Richard Kay, of New York, for the year 1889, page 83, under No. 1292. Usually the spindles graded from 0.9400 to 1.0000 and from 1.0000 to 1.0700 will answer for wine analysis.

I would recommend the continuance of existing methods for another year without change.

The PRESIDENT. Are there any papers to be submitted on this subject? If not, remarks are in order.

The SECRETARY. In regard to the methods of analysis I have addressed a letter to Mr. Colby, calling his attention to the fact that the methods as given are very incomplete and need rewriting, in order that they may be more definite in their directions. In many cases the printed directions refer to such and such a method without describing where it can be found. We have found that in using these methods it was necessary for us to look up all these matters. I therefore asked Mr. Krug to rewrite the methods substantially as they are now, with the introduction of these omissions, so as to make them more definite. I did not have time to submit this draft to Prof. Colby, but he says he hopes the association will take favorable action, and therefore I suggest that these methods be referred to the committee. I think the committee will decide that we had better have these methods as rewritten and very much improved in phraseology and with the omissions I have mentioned filled in, so that anyone can use them without the necessity of consulting any books or papers.

RESUMÉ OF CHANGES IN METHODS OF ANALYSIS FOR FERMENTED LIQUORS.

Many immaterial changes have been made in the wording of the methods, so as to make the processes involved more explicit. The important changes and modifications, which have all been made in strict accordance with the methods now officially adopted in several European countries, will be given in the same order as the methods involved.

Extract.—The first of these is in the manner of determining the extract. Three direct methods are given, one for dry wines and two for sweet wines. Of the latter two the second is described because it is generally preferred by German chemists, permitting them to work with a larger quantity of wine and resulting in a film on evaporation, which is thin and dries much easier than the thick, sirupy residue obtained on evaporating a sweet wine directly.

There seems to be great uncertainty among chemists with regard to the table to be used when the extract is determined by the alternate method. In the table appended on page 21 it is evident that Hager and Schulze differ very materially. Skalweit, in Hannoversche Zeitschrift, Wider die Nahrungsfälscher, states that Hager's numbers are more adapted to extract poor dry wines, while Schulze's agree better with wines having a high extract. He claims that by striking an average between the two we obtain values which agree quite satisfactorily with the results obtained by the direct methods. I have only applied this recommendation once, viz, in the analysis of the official sample, and found his statement correct as far as that wine was concerned.

Total acidity.—In the Official method for total acidity the fact that some litmus solution must be added to white wines so as to furnish an indication of saturation is not mentioned. This has been introduced in the revised method, as well as the value of the reagent used in terms of tartaric acid. The amount of wine to be used is stated.

Volatile acids.—The method for volatile acids has been made more explicit, and the value of the alkali solution in terms of acetic acid stated.

Glycerol.—A number of changes have been made in the official method for glycerol. In the old method for dry wines the bulletin says in the second and third lines, "and then evaporated to dryness." This is radically wrong, as it may entail a loss of glycerol by volatilization. It has been changed to, "and the evaporation carried almost to dryness." Further, the method states to extract once with boiling 90 per cent alcohol and evaporate the filtrate again to dryness. In fact the pasty mass has to be extracted several times, the extracts united, the alcohol partially driven off over a flame, and finally on the steam bath until the residue becomes sirupy. Further, the method does not mention the washing of the sticky precipitate obtained on addition of the ether, with a few cubic centimeters of a mixture of 1 part alcohol and 1.5 parts ether. All these modifications have been introduced.

Reducing sugar.—The old Fehling-Soxhlet method described in the bulletin, as well as the Allihn method, is given. In both cases the reference for the reagents is given. On page 208, second line from bottom of page, is the statement "25 cc. of wine, as in preliminary test," etc. This is no doubt a typographical error and should read 25 cc. of the copper solution. The Allihn method is used altogether in our laboratory, and is much preferable to the other, being not so wasteful in reagent, much quicker, and more accurate. Besides, the results are stated just as in the first method, in terms of dextrose (see Bull. 38, p. 182, sixteenth line from bottom of page). In "preliminary test," "evaporation to two-thirds" is changed to "one-third."

Polarization.—This whole paragraph has been rewritten. The methods are more fully described. The manner of stating the results is given. The length of tube has been changed from 200 to 220 mm., which is the length officially adopted in Europe.

This will make our results comparable with those of the foreign chemists. The equivalents of the degrees of the various instruments are given. A separate method is described for the direct polarization of sweet wines, as these generally have such a high polarization that they are difficult to read when almost undiluted. The methods for inversion and fermentation and the alcohol method for wines rotating less than +0.8° and more than +0.3° Wild after inversion are stated.

The application of the analytical results and the various ways of treating wines which are either non, dextro, or levo, rotatory are fully described.

On page 209 under polarization, paragraph 3, is sheer nonsense, as will become evident to anyone that reads it carefully. It is without doubt a conglomeration of paragraph 3 and the missing paragraph 4.

Tannin and coloring matter.—The composition of the reagents and manner of preparing the boneblack is stated. The name of the method is changed to Löwenthal-Neubauer, as Löwenthal was the originator and Neubauer subsequently modified it. In the first line "lost weight" is changed to "original volume." The titration of the indigo solution is omitted, as it is a constant factor in both titrations. The manner of determining the value of the permanganate solution is also stated. The length of time that the wine must stand with boneblack is given.

Potassium bitartrate.—Although the estimation of the potassium bitartrate is not required in the judgment of wine, the method of estimating it has been introduced, as it is made necessary when it is desired to estimate the tartaric acid by the modified Berthelot-Fleury method.

Tartaric, malic, and succinic acids.—The wording of this method has been changed slightly. The bulletin fails to state how long the lead sulphide should be washed with hot water. This is changed to "washed with hot water until the filtrate shows only a slight acid reaction."

"An excess of $Ca(C_2H_3O_2)_2$" is changed to "an excess of a saturated solution of calcium acetate." In the sentence "This precipitate is dissolved in a minimum of hydrochloric acid, washed with hot water," the words "filtered and the filter" are introduced between "acid" and "washed."

Determination of tartaric acid by modified Berthelot-Fleury method: The method in the bulletin contains an error in stating in the second line "and one-fifth of the mixture, 10 cc., is taken." This is changed to "and to one-fifth of the volume, corresponding to 10 cc. of wine."

A second method for determining the tartaric acid in the filtrate from the potassium bitartrate is given.

Coloring matters.—Methods are described for fuchsin, orseille, and sodium rosaniline sulphonate. The bulletin says in the last case, by shaking wines before and after saturation with ammonia. Kayser's method states to acidify with acetic acid and extract with amyl alcohol.

Ash.—The method is changed to read "The residue from the direct extract determination is incinerated, etc."

Sulphuric acid.—The amount of wine to be used in this method is stated.

Calcium and magnesium.—This method is changed in the manner of treating the ash. Instead of fusing with sodium hydrate and potassium nitrate, it is dissolved in hydrochloric acid. The method of determining these constituents is given more fully.

Phosphoric acid.—Stated as a separate paragraph, the amount of wine and method of treating the ash being described.

Potash.—The Kayser method is described in full.

Sulphurous acid.—Iodine solution is substituted for bromine and standard sodium. Thiosulphate solution is used to titrate the excess of iodin, this method being quicker than the Gravimetric given in the bulletin.

Salicylic acid.—A method for detecting this preservative is given.

Mr. VAN SLYKE thought reference to the committee hardly necessary, as no member of it was likely to know anything about it as compared with the Secretary. He moved that the report and methods, with the changes in phraseology as indicated by the Secretary, be accepted.

Carried.

Mr. HUSTON. It seems to me it might be well to add to this matter of fermented liquors the question of distilled liquors. It is not a simple matter to find a method for the examination of distilled liquors, yet many municipal and State chemists are compelled to examine distilled liquors. I suggest that the field be widened to include distilled liquors, and therefore move that the work of the reporter be made to include fermented and distilled liquors.

Adopted.

The committees on method for potash and nitrogen analysis announced that they were ready to report. The President called for the report of the committee on methods for determining potash.

Mr. WHEELER. The committee reports, on page 178, that section 4 be changed in such a way that the heading will read, "Method of solution for organic compounds;" the last line be changed to "Proceed then as in the Lindo-Gladding or Alternate method;" and that the whole section be transferred to page 179 immediately preceding the word "factors."

The other recommendation is in the fourth line from the top, on page 179: "If the sample has;" at this point insert "over," and strike out the words "to 15."

Mr. FREAR. It seems to me that the committee has failed to understand the instructions of the association in reference to the preparation of a separate statement of the method for solution of the sample. As I understand it, it was made mandatory upon the committee to prepare a simple formula and not the recommendation of a single individual of the association. Now, while the difficulty suggested in the case of section 4 is obviated by the method the committee has adopted, it has not done as the association instructed in the preparation of a separate statement of the method of solution which will answer for both methods.

Mr. WHEELER. The mover of the motion was called out and expressed himself as fully in accord with this change, because otherwise it seemed that the whole matter of potash would have to be rewritten and made over, and that was something it seemed unwise to do.

Mr. FREAR. I still believe that the matter will be presented more logically if the method required by the motion is followed. I do not believe, as the committee seemed to infer, that it would require any extensive rewriting.

Mr. ROSS understood that the committee was also to make some provision in its report for mixed fertilizers containing potash in organic compounds.

The PRESIDENT had understood that the matter was to be revised in the line suggested by Mr. Ross.

Mr. WHEELER. I think there was some difference of opinion in the committee itself. The result as reported was the resolution of the committee, not any individual opinion.

The question was put and the report adopted, 10 to 1.

The PRESIDENT. If you desire to further charge the committee you can do so.

Mr. FINNEY wanted to know what was meant by "etc." in the words "tobacco stems, cotton-seed hulls, etc." Did it include cotton-seed meal?

Mr. WHEELER, called upon by the President for enlightenment. He thought an organic compound was something produced in the animal or vegetable economy.

In defense of the report, Mr. Wheeler added that a number of chemists present wanted to ignite in the case of mixed fertilizers which contained such substances and others who did not want to ignite. One party wanted the committee to put it one way, the other in another way.

Mr. FREAR. My understanding was that it was clearly indicated this morning that we wanted instructions for both—in one case to be clearly indicated as total, in the other as water-soluble potash—simply in the preparation of the sample. It is obvious we can not use the same method of preparation in such samples as these and get the same result in these two cases. This makes all the more emphatic the need for separation of the instructions in regard to the preparation of solutions.

Mr. PAYNE moved a reconsideration of the previous action in order to decide the matter one way or the other.

The PRESIDENT did not think reconsideration was required; his recollection was that "etc." included cotton-seed meal. If we want a change to cover the total potash question we will have to have an entirely different arrangement for making solutions.

The report of the committee on methods for analyses of nitrogen was called for.

Mr. Bartlett's first recommendation was page 173, paragraph 2, instead of "standard ammonia" put "standard alkali."

Mr. HUSTON. Under the rules of this association the rest of this paragraph is stricken out. The rule adopted two or three years ago says that explanatory insertions are to be carried one year and then dropped.

The second recommendation was on page 174, first line of paragraph, "Manipulations" change 0.8 gram to 3 grams.

Third recommendation, in the third line of same paragraph, 22 cc. to be changed to 20 cc.

Fourth recommendation, page 175, change the word "thoroughly" after the word "shake" in the third line to "until thoroughly mixed with acid." The punctuation in the same line to be changed, putting a comma after "thoroughly," a semicolon after "thiosulphate," and a comma after "salicylic acid" in the fourth line

The reporter suggested that next year the method presented this year by Dr. Fassbender, for determining nitrogen in the presence of nitrates, be given a trial.

Mr. McDonnell thought that in some cases 0.7 gram of material might be found too much. The correction of typographical errors, he thought, should be left to the secretary.

Mr. de Roode. Mr. Huston has stated that it is the custom for the explaining portion of a paragraph to be dropped out. I think we ought to leave in "one-tenth normal alkali solution * * * is recommended."

Upon motion the recommendation was adopted.

In discussing the second recommendation, Mr. Huston moved that in place of 3 grams the number 3.5 grams be substituted in all cases where a given substance is taken.

Mr. Peter. The reason why 1.4 was taken is that you can obtain correct results with that much sodium nitrate, which is about the richest nitrate likely to be analyzed, using the portions of the reagents here specified; but if you take larger quantities of sodium nitrate you will not have enough reagents. I should not like to increase these quantities without some rule by which to know when larger quantities are to be taken.

Mr. Huston's amendment was voted upon and adopted.

The third and fourth recommendations were adopted without discussion.

Some discussion arose on the fifth recommendation as to whether the addition of potassium sulphate in the Gunning method caused foaming.

Mr. Bartlett thought not; it was caused, he stated, by the way the heat was applied.

Mr. Ross said that several German chemists reported great trouble from foaming where excessive quantities of potassium sulphate were used; they added only a comparatively small quantity at first, and then gradually the full amount required.

Upon motion the recommendation was adopted.

Mr. Bartlett stated that the association has no Official method for determining nitric nitrogen. He desired that the reporter for next year be recommended to investigate the matter.

Mr. de Roode moved that the Tiemon-Schulze method be printed as a provisional method until finally adopted.

Mr. Winton moved to amend that by having the Tiemon-Shulze method printed at once as official.

Mr. Frear moved that the matter be referred to the committee, which was carried.

Mr. Peter moved that the Ruffle method be stricken out from the methods of the association.

Mr. **Frear** thought it desirable to retain it as a check method.

Mr. **Carpenter** had had very satisfactory results with the Ruffle method and disliked to see it stricken out.

The matter was referred to the committee on methods.

The president then called for the report of the committee on nominations, which was as follows:

For president, H. A. Huston, State Chemist of Indiana.

For vice-president, B. B. Ross, State Chemist of Alabama.

For secretary, H. W. Wiley, Chief Chemist of U. S. Department of Agriculture.

Additional members of executive committee: E. H. Jenkins, Chemist and vice-Director Connecticut Experiment Station; F. W. Woll, Assistant Chemist Wisconsin Experiment Station.

The report of the committee was adopted and the secretary instructed to cast the vote of the association for these officers, which was accordingly done.

The president then called for the report on cattle foods, which was read by Mr. Patterson.

REPORT ON THE INVESTIGATION OF METHODS FOR THE ANALYSIS OF CATTLE FOODS.

By H. J. Patterson.

On November 28, 1893, circular letters were sent to the experiment station chemists and others likely to be interested in the analysis of cattle foods, asking for suggestions as to the lines of investigation best to be conducted during this year, and soliciting their coöperation in the work. These letters brought replies from twenty-three chemists who were willing and desired to take part in the investigations, and many of them gave suggestions as to the lines and nature of inquiry and the most important points needing immediate reform.

All the suggestions given as to the points for investigation could not be considered, as it would have caused too much work; consequently, only three points of inquiry (fat, crude fiber, and the separation and direct determination of the nitrogen-free extract constituents), which seemed of importance to the greater number, were selected for investigation, and the methods for the investigation along these lines were made up from the suggestions received from the different chemists. No one chemist was asked to do all the work proposed, but only that part which was of most interest to him.

The greater number of chemists who agreed to take part in the work found it impossible to do so, only nine chemists reporting the results of work done. While we all know that the demands upon the time of the station chemists are in most instances exceedingly varied and onerous, yet it is to be regretted that more of us can not take part in this work, as the value of our association work rests to a very great degree in the number included in the averages. While all of us have many problems confronting us, and are working on subjects of practical interest to the farmer, and things that come in direct touch with them and their operations, yet we must not for a moment think that the study of methods is a waste of time and money or give them but secondary consideration.

In reference to cattle foods, the improvement of our methods is of far greater value than the analysis of any number of materials by our present methods. If we are to grapple intelligently with the subject of feeding, and make any marked advance in our knowledge of the subject, we must make radical changes in our methods, and prepare to soon give up the present arbitrary and erroneous classification and division.

With these considerations, together with the general feeling and expression of chemists on every hand, I believe it is full time to concentrate our efforts on the separation and determination of the constituents of a single class of ingredients rather than expend any more effort on perfecting the present arbitrary method of analysis of cattle foods.

MOISTURE.

The moisture was not determined as a matter of investigation of methods, but simply to serve as a check and to enable the results of the other determinations to be calculated to a water-free basis. The results of these determinations are given here as a matter of record.

Moisture determinations.

	Sample 1, corn stover.	Sample 2, wheat.	Sample 3, cotton-seed meal.
	Per cent.	*Per cent.*	*Per cent.*
1. U. S. Department of Agriculture, W. H. Krug	6.85	12.64	8.57
2. Texas station, P. S. Tilson	[2]1.38	[2]3.36	[2]2.20
3. Massachusetts station, C. H. Jones	6.48	12.28	8.38
4. New Jersey station, L. A. Voorhees	7.04	12.96	8.23
5. Germany, Dr. Fassbender[1]	[2]5.60	[2]11.15	[2]7
6. Maryland station, H. J. Patterson	6.83	12.21	7.95
Average	6.80	12.52	8.28
Greatest variation from average	.32	.44	.33
Greatest variation from average in per cent	4.7	3.5	4
Difference between highest and lowest	.56	.75	.62

[1] Dried for 3 hours.

[2] Excluded from averages.

The above results of moisture determination are interesting from the fact that they show that by the use of the official methods the results are remarkably close, and that the percentage of variation from the average is growing constantly less each year. These moisture determinations would seem to indicate that, in experienced hands and with a due amount of care, the present Official method for the determination of moisture was quite, if not entirely, satisfactory.

ETHER EXTRACT (FAT).

The following is a copy of the instructions sent out for the study of methods for the determination of fat in cattle foods. The samples to be used were: No. 1, corn stover; No. 2, wheat meal; No. 3, cotton-seed meal.

1. The Official method.
2. Dry the sample for thirty-six to forty hours over sulphuric acid in a vacuum at a temperature of 65 to 70° C., and extract as in the Official method.
3. Proceed as in the Official method, except using the petroleum spirit sent out with the official samples instead of ether.

Method 2 was incorporated at the suggestion of Dr. Wiley, and as his letter expresses the value and reason for such a manner of procedure so clearly, I will quote therefrom:

"I should like to see some work on the determination of moisture by drying for three or four days over sulphuric acid, or other water-absorbing material, at a temperature not exceeding 60 or 70° C., and practically in a vacuum. My impression is that it is not so much the drying in air as it is the high temperature which causes the insolubility of the glycerids when subjected to the action of ether or petroleum.

"I think one of the great efforts which should be made by our association is to make the methods of analysis as simple as possible. If drying at a low temperature, for instance, in the circumstances mentioned will take out all the moisture and not affect the solubility of the glycerides, it would be a simpler way of doing it than by drying in hydrogen."

None of the reports gave results with method two. My own results with this method, as will be seen by the following table, agree very closely with those by the Official methods, both for fat and moisture. The samples, during the long drying, blackened on the surface considerably, but I believe this would be obviated by heating either a shorter time or at a lower temperature.

The results as given in the table for fat determinations show that there has been no improvement in the closeness with which different analyses work in determining fat. The average results with ether and petroleum agree very well. The results by petroleum extract in the case of the corn stover (sample No. 1) are markedly close and would seem to indicate that for such substances the petroleum would be preferable.

These results, as a whole, show that before any definite change from our present method can be recommended further tests of these points must be made.

Fat determinations.

[Results calculated water-free.]

	Sample No. 1.		Sample No. 2.		Sample No. 3.	
	Ether.	Petroleum.	Ether.	Petroleum.	Ether.	Petroleum.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1. Texas station, P. S. Tilson	[1]3.02	....	2.72	....	13.49	....
2. New Jersey station, L. A. Voorhees	1.60	1.17	2.29	2.16	13.30	13.01
3. Massachusetts station, C. H. Jones	1.78	1.35	2.45	2.82	13.59	13.86
4. Maryland station, H. J. Patterson	1.58	1.38	2.19	2.00	13.09	12.86
5. U. S. Department of Agriculture, W. H. Krug	2.01	....	2.57	....	13.59	....
6. Germany, Dr. Fassbender	1.56	....	2.18	....	12.98	....
Averages	1.71	1.30	2.40	2.33	13.34	13.24
Greatest variation from average	.30	.13	.32	.49	.36	.62
Greatest variation from average in per cent	17.50	10.00	13.30	21.00	2.70	4.60
Difference between the highest and lowest	.45	.21	.54	.82	.61	1.00
Extraction without drying.						
Maryland station, H. J. Patterson	2.41	....	2.42	....	13.78	....
Drying in a vacuum at 65° to 70° for forty hours before extraction.						
Maryland station, H. J. Patterson	1.55	....	2.11	....	13.16	....
Moisture lost in drying in the vacuum over sulphuric acid at 65° to 70° C. for forty hours.						
Maryland station, H. J. Patterson	6.30		13.02		8.20	

[1] Excludes from averages.

CRUDE FIBER.

1. Test Official methods, using 1¼ per cent H_2SO_4 and NaOH solution, using samples Nos. 1, 2, and 3.

2. Test Official method, using 2½ per cent H_2SO_4 and NaOH solutions, using samples, Nos. 1, 2, and 3. The reason for using all the samples is to try and determine if the ingredients, not starch, are not as likely to protect the fiber and be as hard to dissolve as the starch is.

3. Take 2 grams substance; place in a strong flask; add 200 cc. of boiling 1 per cent HCl. Cork the flask tightly and place in a water bath heated to 95 to 98° C., and maintain at this temperature for two hours; filter, wash, and then bring back into the flask with 200 cc. boiling 1 per cent Na_2CO_3. Cork tightly, and place in the water bath at 95 to 98°, and maintain this temperature for two hours. Filter, wash, and proceed as in the Official method. In placing the bottles in the bath be sure the level of the water in the bath is above the level of the liquid in the bottles. Shake the bottle every ten minutes during digestion.

NOTE.—In all cases test for the presence of starch and pentosans, and determine the amount of nitrogen remaining.

The samples used were the same as used in the fat determination.

Crude fiber determinations.

[Results calculated water-free.]

	Sample 1.			Sample 2.			Sample 3.		
	1.25 per cent H_2SO_4 and NaOH.	2.5 per cent H_2SO_4 and NaOH.	1 per cent HCl and Na_2CO_3.	1.25 per cent H_2SO_4 and NaOH.	2.5 per cent H_2SO_4 and NaOH.	1 per cent HCl and Na_2CO_3.	1.25 per cent H_2SO_4 and NaOH.	2.5 per cent H_2SO_4 and NaOH.	1 per cent HCl and Na_2CO_3.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1. U. S. Department of Agriculture, W. H. Krug	35.42	32.95	39.32	2.37	2.01	2.86	4.34	3.99	4.08
2. Texas, P. S. Tilson	33.43	....	....	2.11	....	....	3.99	....	....
3. New Hampshire, F. D. Fuller	31.74	29.33	38.72	2.13	1.75	3.25	3.23	2.87	4.78
4. Germany, Dr. Fassbender	36.70	....	....	2.67	....	....	5.02	....	....
5. Scotland, Dr. Aitken	34.36	30.90	35.95	2.46	2.04	3.10	4.11	3.55	7.40
6. Maryland, H. J. Patterson	37.56	34.72	50.33	2.29	2.02	3.54	4.10	3.87	5.83
7. Wisconsin, F. W. Woll	33.69	31.07	35.23	2.36	1.89	2.67	3.73	3.29	7.69
Averages	34.70	31.79	37.30	2.34	1.94	3.08	4.05	3.68	5.96
Greatest variation from average	2.96	2.93	2.07	.33	.19	.78	.32	.39	1.88
Greatest variation in per cent	8.5	9.2	5.5	14.1	9.8	25.3	7.8	10.06	31.3
Difference between highest and lowest	5.82	5.39	4.09	.56	.29	.87	.61	.70	3.61

Crude fiber by method 3, W. H. Krug, analyst.

	Flask open.	Flask closed.
	Per cent.	*Per cent.*
Sample 1	36.84	39.32
Sample 2	3.18	2.86
Sample 3	5.52	4.08

The results of the crude fiber determinations are fully up to the standard, and on the whole may be regarded as an improvement, but yet they are far from being satisfactory.

The results do not show any real reason for the use of the two different strengths of solutions as indicated in the Official method, and there is nothing whatever that can be used as a guide and tell when to use one strength or when to use the other. Consequently the decision must be an arbitrary one, suiting the caprice or convenience of the analyst.

The results of this year's work, on the whole, seems to favor the 1¼ per cent solution as being most uniform, and coupling with this the fact that all the starch seems to be dissolved by the 1¼ per cent solution, and also the fact that this is the strength most universally used and the strength with which the greater number of fodder analyses have been made, I would therefore recommend that the association method authorize only one strength of solution for crude fiber determinations, and that as far as we know the 1¼ per cent solution would seem to answer our purpose best.

The results with method (3), though not agreeing very well among themselves and not at all with the others, yet I think they are encouraging in that they indicate that the imitation of the solution and processes of animal digestion, and that the use of a fixed temperature in closed vessels rather than the uncertain temperatures and strength of solutions obtained in boiling in open vessels, is a very fruitful field for investigation along the line of crude fiber determinations. I therefore commend this phase of the question to the association and the next reporter.

NITROGEN-FREE EXTRACT.

In response to the very general demand and numerous suggestions from members of the association a schedule was made for work along the line of the separation of the substances which go to make up the nitrogen-free extractive materials and for their direct determination.

Appreciating the fact that the majority of agricultural chemists want, and will have, simplicity in their everyday methods, and also the fact that many of us do not have much money to expend for apparatus that would not be useful unless it could be or was to be used in the Official methods, the tentative schedule outlined for the nitrogen-free extract work was not made to conform in all cases to the latest methods and the methods which have given indications as being the most accurate. In a few instances the methods outlined were made to conform with the methods already used in the investigations which some of our experiment stations have carried on. These are the reasons which backed and caused the adoption of the schedule for this work which was sent out. The work outlined considered the dextrose, the sucrose, the starch, the dextrin, and the pentosans.

In starting in upon this line of inquiry it was not expected that much would come of it the first year; or, in fact, that we would be able to definitely narrow this general dumping ground in the least by a single year's work. Yet it was hoped that starting this work officially would excite some individual work along the line and be fruitful enough to at least give indication of how to proceed by another year.

This much, I think, can be truthfully said to have been accomplished, for I have now in my possession a paper to be read to you upon the determination of starch in feeding stuffs; and the results of the association work, though small in amount, I believe will give fruitful indications for future work.

Only four of us found time to do any work on the nitrogen-free extract question, and that was confined in most instances to a single sample. Consequently there is not sufficient data to make an intelligent discussion of the subject, or upon which to make any definite recommendations to the association to give its time to a discussion of the question; yet the results are valuable, and give good indications as to how to proceed in our future work. It will probably be interesting in this connection for the association to know that the results obtained so far, while not as good as desired, yet are fully as good and in some instances better than the first work of the association on butter, reverted phosphoric acid, moisture, fat, or crude fiber. I therefore recommend that the association refer this matter with the data of this year to the next reporter, with the request that this subject be given first place and made the most important point for the next year's investigations.

ANALYSES OF CATTLE FOODS BY DR. STROHMER.[1]

The three samples of cattle foods sent by the reporter were analyzed by Dr. Friedrich Strohmer with the following results. The cane sugar, invert sugar, starch, dextrin, and crude fiber were determined by the methods forwarded with the samples:

Results.

	Sample No. 1.	Sample No. 2.	Sample No. 3.
Cane sugar	1.92	3.21	1.73
	1.84	3.30	1.84
Invert sugar	1.86	.54	.00
	1.51	.65	.00
Dextrin determined by precipitation with ammoniacal lead acetate	3.07	8.04	6.44
	3.87	10.05	5.18
Dextrin by method (2)	4.12	9.15	5.80
	3.50	9.96	5.45
Starch	15.64	50.30	.00
	15.90	50.10	.00
Indigestible fiber by 1¼ per cent sulphuric acid and soda lye	37.42	2.57	7.35
	37.58	2.45	7.49
Indigestible fiber by 2½ per cent sulphuric acid and soda lye	31.03	2.12	5.97
	30.85	2.03	5.90
Indigestible fiber by 10 per cent hydrochloric acid	30.80	1.95	5.60
	....	....	....

Dr. Strohmer says, in speaking of the above results:

"In respect of the methods used for cattle food analysis we must express our judgment that they are in no sense simple, and also the agreement of the results, in spite of the duplicate analyses carried out at the same time and in the same manner with the greatest care, is not satisfactory."

The PRESIDENT. If there is no discussion of the report it will be referred to the committee in the regular way. Any further papers are now in order.

Mr. Patterson then read a paper by Mr. Stone, of Purdue University, and Mr. Krug a short paper on cattle foods.

A COMPARISON OF METHODS FOR THE DETERMINATION OF STARCH.

By W. E. STONE.

There is no longer much doubt among chemists that, in food analysis, the present practice of classifying a large number of widely varying substances under the head of nonnitrogenous extract matter as an homogenous material is wholly erroneous and misleading. The variety of substances covered by this term is well known, and while it is granted that they are mainly of carbohydrate nature, it is equally clear that, so far as food valuation is concerned, there should be some degree of differentiation between sugars, starches, gums, and the more or less soluble forms of cellulose.

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[1] These analyses came too late for incorporation in the report of Mr. Patterson.

The position of the pentosans in this class of bodies has been pointed out in its different aspects by myself and others at intervals during the past four or five years. It has been shown that these bodies form a distinct constituent of the nonnitrogenous-extract matter to a greater or less degree in all fodder articles; that under present analytical methods their individuality as food constituents is wholly merged into that of the more valued carbohydrates; some light has been thrown upon their digestibility, and analytical methods of a fair degree of accuracy have been devised for their estimation in the presence of other similar bodies.

Mention is made of this instance here to show that really some progress has been made, and that, too, within a short time in our knowledge of what until recently has been an obscure constituent of the nonnitrogenous-extract matter.

The object of this present paper is to call attention along the same line to certain proposed methods for the quantitative estimation of probably the most important of these bodies, viz, starch. At first thought it may appear that the existing methods for the determination of starch are sufficiently satisfactory and accurate; and this is doubtless true so far as starch alone is concerned. But it must be remembered that starch almost always occurs in connection with other soluble nonnitrogenous matters, under which conditions its estimation becomes quite another matter and constitutes exactly the question under discussion. The pentosans or gums are doubtless the most common of these accompanying substances and unfortunately for the analyst behave toward many analytical methods precisely like starch. Hence, a result which is interpreted to mean starch by a strict adherence to method may be due in a very large degree to other substances. For example, brewers' grains which, from the very process through which they have passed, contain little or no starch, will, by the ordinary method of inversion and titration, be credited with a large amount of starch. Again, hay or straw yield considerable amounts of reducing sugars on inversion, although they contain a minimum of starch or even none at all. In both these cases the results are due to the inversion of insoluble gums which are present in large quantities.

With these facts in mind it is apparent that a food analysis should give some information with regard to the relative amounts of these different substances present, and should include determinations of the pentosans, sugars, and starches separately.

In this connection the inquiry arises as to how accurately it is possible to determine these bodies as they occur in connection with each other in the ordinary feeding stuffs by current and accepted methods. As regards the pentosans and sugars, the state of our knowledge seems far more satisfactory than in the case of the starches, in proof of which proposition the following data are offered.

Five methods of starch determination have been compared upon a variety of materials. Four of the methods involve the inversion of the starch and the estimation of the sugar thus formed by Volumetric or Optical methods; the fourth method is based upon a combination of the starch with one of the alkaline earths. Following are the details of the different methods:

1. Inversion of the starch with hydrochloric acid and titration with Fehling's solution. Known as Sachsse's method. Three to 5 grams of the starch-bearing material are heated in a water bath with 200 cc. of water and 20 cc. of concentrated acid, during three hours; the acid is neutralized with soda, the solution filled to 500 cc. and titrated with Fehling's solution in the usual manner.

2. Inversion with nitric acid and polarization. Proposed by Guichard (Bull. d. l. Soc. Chim. d. Paris (3), 7, 554). Three grams of the material are heated in a water bath during one hour with 100 cc. of 10 per cent nitric acid, filled to 200 cc. and polarized. The readings are calculated to dextrose.

3. A modification of the preceding. Three grams of material are heated in the water bath with 100 cc. of a saturated solution of oxalic acid during one hour, the object being to bring the starch into solution. The liquid is cooled and filled to

200 cc. with 10 per cent nitric acid, filtered, and the filtrate heated in the water bath one hour, attached to an inverted condenser. The solution is then polarized. This method is preferable to the other in that the cellulose and other insoluble substances are removed from the influence of the nitric acid.

4. Inversion of the starch by salicylic acid and polarization. Proposed by A. Baudry (Zeit. f. Spiritus Industrie, 15, pp. 41 to 42). A normal weight (5.376 grams) is adopted for the Laurent polariscope. This weight or some multiple of it is placed in a 200 cc. flask with 100 cc. of water and 0.4 to 0.5 gram of salicylic acid and heated in a water bath for thirty minutes; fill to within 20 cc. of the mark with hot water, cool rapidly, clarify by adding a few drops of ammonia, filter, and polarize in a 400 mm. tube. The readings on the percentage scale indicate percentages. In our practice sodium hydroxid was used instead of ammonia for clarifying.

5. Precipitation of the starch from the condition of paste by barium hydroxid and determination of the excess of the latter by titration with a standard acid. Proposed by A. von Asboth in 1887. One gram of the material, from which the fat has been extracted, is boiled with water to convert the starch into a paste; 25 cc. of a solution of barium hydroxid of known strength is run in and the whole allowed to cool; then add 100 cc. of 85 per cent alcohol and fill to 200 cc. A compound of starch is formed containing 19.1 per cent of BaO, which is precipitated by the dilute alchohol. The solution is filtered and the excess of barium hydroxid is determined by titrating aliquot portions with standardized hydrochloric acid, using phenolphthalein as an indicator.

The materials to which these methods were applied included eight different samples, as follows: Pure potato starch, wheat bran, wheat middlings, wheat flour, dried potatoes, corn meal, hay, cotton-seed meal, and a mixture of starch, sugars, and dextrin. Four of these were samples sent out by the reporter on feeding stuffs of the Association of Official Agricultural Chemists. Before examination the samples were extracted with water and ether to remove soluble carbohydrates and fats.

The results obtained are best shown by tabulation, as follows:

	Inversion by HCl.	Inversion by HNO_3.	Solution by oxalic acid. Inversion by HNO_3.	Inversion by salicylic acid.	Precipitation by $Ba(OH)_2$.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1. Potato starch	85.75	85.50	85.75	85.47	85.58
2. Dried potato	70.92	69.79	68.53		64.25
3. Wheat flour	77.69	70.65	65.29	69.38	59.76
4. Corn meal	73.24	66.81	70.55		62.11
5. Wheat bran	65.86	40.25	38.68		70.77
6. Hay	5.48	19.10	19.10		66.47
7. Wheat middlings	30.00	63.09	60.24		60.44
8. Cotton-seed meal	4.15				54.65
9. Starch mixture	9.58	21.00	24.08	18.80	33.99

From a survey of these data it appears that any of the methods employed give satisfactory results when dealing with starch alone. It is important to recognize this in order that the responsibility for the discrepancies in connection with the other materials may be properly placed where it belongs, viz, that the variations are due to the complex nature of the materials and not to the method. Allowing for moisture and cellulose, the starch was pure and each method gave credit for the entire amount present. Turning to the other materials, the following is to be noted:

1. Inversion by hydrochloric acid. The starch is converted into dextrose, and under carefully controlled conditions the reaction is quantitative. Wohl has shown that a very small quantity of acid is efficient in producing complete inversion. (Berichte d. D. Chem. Gesell., XXIII, 2084.) According to Märcker, the method of

Sachsse here employed gives low results owing to a slight destruction of dextrose by long heating with the acid. This objection does not appear here in comparison with the other methods. There is undoubtedly danger in the other extreme in not heating long enough. The samples numbered 6, 7, 8, and 9 were treated according to the directions sent out by the Association of Official Agricultural Chemists, which call for the removal of heat as soon as the starch-iodin reaction disappears. It is possible that under these conditions, which are accomplished in a very short time, an appreciable amount of starch does not pass beyond the dextrin stage, and escapes detection by Fehling's solution. In this connection it is noticeable that the samples, Nos. 6, 7, 8 and 9, treated in this way gave lower results by this than by the other methods.

2. The methods calling for the use of nitric acid seem unusually destructive, yet in the case of pure starch they have given results fairly comparable with the others. As originally proposed by the author, it was feared that the cellulose would be attacked, and the use of oxalic acid as in the third method was proposed. This provides for the solution and removal of the starch by a comparatively mild reagent, and this solution is then inverted by the nitric acid apart from the accompanying constituents of the material. The method has a certain advantage of ease and rapidity; the solutions obtained for polarization are also beautifully clear, and admit of accurate readings. The effect of the direct action of the acid in the second method is not so marked as might be expected. In the case of cotton-seed meal the solutions were distinctly levorotatory, and no attempt was made to calculate to starch or dextrose.

4. Both salicylic and benzoic acids completely dissolve starch; the solutions have a righthanded polarization and the degree of rotation is directly proportional to the amount of starch present. The method was originally proposed for the valuation of commercial starches, and with such materials is fairly accurate. With feeding stuffs, however, the resulting solutions are frequently too highly colored and opaque to admit of examination in the polariscope. The method is therefore of very limited application.

5. The use of barium hydroxid as a means of combining and precipitating starch from solution has attractive features and has been both highly praised and severely criticised. It necessitates the previous removal of all oils from the material. In our hands the results obtained have been somewhat erratic.

One fact is apparent in connection with the above data. If we leave out of consideration the pure starch sample, the results are more or less discordant and in some cases quite unexplainable. For instance, the hay and cotton-seed meal, when boiled with water, do not give the iodin reaction for starch, yet each of these special methods for the determination of starch credit them with from 4 to 60 per cent of the same. Even fifteen minutes' warming with very dilute hydrochloric acid gives an appreciable amount of what we are bound to interpret as starch. This brings us to the inevitable conclusion that other bodies than starch are present and respond toward each of these reactions in the same way as starch does. We know, moreover, that the pentosans are such bodies, and that they are invariably present to a greater or less degree in materials of the kind under examination.

In order to obtain some experimental data bearing upon the subject, portions of an isolated sample of pentosans were subjected to each of the methods under discussion. The material was, properly speaking, xylan, since on inversion it yields only xylose. It was obtained from wheat straw by boiling the same with lime water and precipitating the xylan with alcohol. It possessed as high a grade of purity as has thus far been attainable in the preparation of these materials. It was free from any other form of carbohydrate, and contained about 6 per cent of ash materials.

One gram of xylan heated for thirty minutes with 50 cc. of water and 4 cc. of strong hydrochloric acid was entirely dissolved. The solution was neutralized and made up to 100 cc. Of this, 6.7 cc. were required to reduce the copper from 10 cc. of Fehling's solution, a result equivalent to 67.16 per cent of starch.

One gram of xylan was heated with 50 cc. of 10 per cent nitric acid for one hour and became completely dissolved. The solution was made up to 100 cc. and polarized. The reading was equivalent to that produced by 44.73 per cent of starch.

One gram of xylan was treated with 50 cc. of a concentrated solution of oxalic acid for one hour, by which it became completely dissolved. Made up to 100 cc. with 10 per cent nitric acid and heated another hour, the polariscope readings were the same as in the previous case, equivalent to 44.73 per cent starch.

The salicylic-acid method was not applicable.

One gram of xylan was boiled with 30 cc. of water, 25 cc. of barium hydroxid solution was added, and afterwards 35 cc. of 85 per cent alcohol, and the whole made up to 100 cc. with water. Upon titrating the excess of barium hydroxid, an equivalent of 47.48 per cent of starch was indicated.

These results show conclusively that the pentosan which is most characteristic of feeding stuffs, and which has been shown to occur in all such materials, behaves toward the reagents named in precisely the same manner as starch, and in a no less marked degree. The conclusion is unavoidable that none of the methods for determining starch, based upon the above principles, can be employed with any degree of accuracy upon grains or forage plants or any materials where the presence of these pentosans is probable.

The most hopeful way of avoiding these difficulties would seem to be some method of bringing the starch into solution and removing it from its accompanying carbohydrates without any corresponding influence upon them. One method seems to offer this advantage, viz, the application of diastase or malt infusion to the starch containing material. This method is recommended for use among the European experiment stations; it has been shown to yield accurate results with pure starch and can be objected to, if at all, only on account of length of time required and the difficulty of always having the proper preparation of diastase or malt infusion at hand. These are, however, minor objections in the light of greater accuracy secured. The general features of the method are as follows: The weighed material is boiled with water (most effectively under pressure) to convert the starch into a paste; after cooling to 65° C. a small quantity of malt infusion is added and the temperature maintained at 60° to 65° for one-half hour. The starch reaction having disappeared the solution may be filtered off, washing the residue thoroughly, the filtrate warmed with a few cubic centimeters of acid to complete the conversion into dextrose and finally neutralized, made up to definite volume and titrated. It remains to be seen what the effect of this process may be upon the pentosans or other similar bodies, concerning which there are few data at hand. I have made some preliminary tests which indicate that these bodies are not affected by diastase. One gram of the xylan already mentioned after boiling with water received 10 cc. of a fairly strong infusion of malt and was kept at 65° C. for one-half hour. At the same time a sample of starch was boiled to a paste and treated with the malt infusion in the same way. The starch reaction disappeared and the solution reduced Fehling's solution strongly, but the xylan did not change the Fehling solution in the least nor did it appear to have been altered.

It seems, therefore, that in this last method the difficulties presented by the more common methods are to be avoided, and with this end in view it is recommended to the attention of those interested in the solution of this important problem.

I am indebted to Mr. D. B. Hoffman for assistance in the preparation of a portion of the analytical data here presented.

AN ATTEMPT TO ANALYZE A MIXTURE OF DEXTRIN, STARCH, DEXTROSE SUCROSE, AND GUM ARABIC.

By W. H. KRUG.

The Association of Official Agricultural Chemists' reporter on feeding stuffs this year sent out a sample composed of the above substances, and designed to represent in a certain measure the nitrogen-free extract of a fodder. The nitrogen-free extract has hitherto been always estimated by difference, and has always been the dumping ground for a class of constituents whose estimation was either impossible under the circumstances or had not yet been attempted. Naturally, methods which are practicable when a mixture of pure carbohydrates is to be analyzed are not necessarily applicable to a nitrogen-free extract, which may contain a number of other substances whose presence tends to vitiate results. Among those may be numbered pectin, the tannins, coloring matter, glucosides, etc. Nevertheless it was hoped that the analysis of such a mixture might lead to some definite conclusions or might at least stimulate other chemists so that this subject, which has been avoided so long and so carefully, might be attacked in the proper manner.

Accompanying this sample were a number of methods which will be found in Mr. Patterson's report. A perusal of the methods submitted showed certain points where they might lead to erroneous results. The following objections were the most important, the numbers corresponding to those of the methods:

1. *Sugars and dextrin.*—It was found impossible to satisfactorily filter off the starch unless some way of entangling it was employed. For this I used alumina cream with good success. When the starch is subsequently inverted with hydrochloric acid and this solution neutralized with sodium carbonate, the aluminum is all precipitated and can be filtered off so that it exerts no influence on the subsequent reduction of the copper solution.

2. *Cane sugar.*—The solution in this case contains not only dextrose and sucrose, but also all the dextrin and gum arabic, both of which are at least partially converted into substances capable of reducing Fehling's solution when treated with hydrochloric acid.

3. *Dextrin.*—Ammoniacal lead acetate will here precipitate not only the dextrin but likewise the gum arabic and probably some if not all of the dextrose and sucrose.

The same objection applies to the second method given for dextrin. Precipitation with alcohol is one of the ways of detecting gum arabic in wines.

I concluded to try the following scheme, which promised to give all the constituents but the dextrin. If no other foreign substance was present this may be estimated by difference.

I.—GENERAL SEPARATION OF CONSTITUENTS.

1. Weigh out 2.5 grams of the substance and digest one hour at room temperature with 50 cc. of water, stirring frequently. Add a few cubic centimeters of alumina cream; filter and wash with cold water until the filtrate amounts to 100 cc. Residue contains the starch.

2. To the filtrate add 2 cc. of basic lead acetate solution; stir and filter into a 250 cc. flask, washing until the filtrate amounts to 240 cc. Precipitate is the lead compound of gum arabic.

3. Make the filtrate up to the mark with saturated solution of sodium sulphate; shake and filter. The filtrate contains the dextrose, sucrose, and dextrin, 100 cc., being equivalent to one gram of substance.

II.–ESTIMATION OF CONSTITUENTS.

1. Starch—a. The starch was inverted in a flask by the method proposed by the reporter.

b. The starch was determined by Märcker's method. This can be found in Märcker, Handbuch der Spiritusfabrikation, fifth edition, page 93, and is as follows:

The starch is gelatinized with 50 cc. of water in the metal cup accompanying the autoclave. It is then cooled to 70° C. and treated for twenty minutes at this temperature with 5 cc. of malt extract (100 grams malt to 1,000 cc. of 20 per cent alcohol). Five cubic centimeters of 1 per cent tartaric acid are then added, the cup covered, placed in the autoclave and kept there for one-half hour under a pressure of 3 atmospheres. After cooling it is again digested at 70° C. for twenty minutes with 5 cc. of malt extract, then washed into a 250 cc. flask, made up to the mark after cooling and filtered. Two hundred cubic centimeters are then inverted for two hours with 15 cc. hydrochloric acid, sp. gr. 1.125. The acid is neutralized with sodium carbonate, the solution made up to 500 cc., filtered, and 50 cc. used for each reduction.

The above proportions and dilutions have to be varied with the starch present in the substance under analysis.

2. Gum arabic.—The lead precipitate containing the gum arabic was placed in a flask with 100 cc. of 12 per cent hydrochloric and distilled in the manner prescribed by Tollens and de Chalmot until no more furfurol came over. In an aliquot portion of the distillate which was neutralized, acidified with acetic acid and made up to 1 liter, the furfurol was estimated gravimetrically by precipitation with phenylhydrazin acetate. From the weight of furfurolhydrazon obtained the gum arabic was calculated in the usual manner.

3. Dextrose and sucrose.—The dextrose was determined in the final filtrate by direct reduction. The sucrose was estimated by the following method:

One hundred cubic centimeters of the solution were placed into a 200 cc. flask and treated in a water bath at 55° C. for five hours with 5 cc. of yeast liquor (one cake of Fleischmann's compressed yeast to 50 cc. of water). The yeast was then killed by boiling, the solution cooled, made up to the mark with a few cubic centimeters of alumina cream, filtered, and the total reduction determined in the filtrate. The increase in copper reduced was due to the invert sugar formed, and from this the sucrose was calculated.

For comparison with the other analysts the sucrose was also determined by investing with hydrochloric acid, as suggested in the methods submitted.

The copper reductions were made by three methods, viz: For dextrose direct, the old gravimetric Fehling method given in Allen's Commercial Organic Analysis (vol. 1, p. 227), and Allihn's gravimetric method, given in Bulletin 38 (Proceedings of the Association of Official Agricultural Chemists, p. 185). For the invert-sugar determination necessary to arrive at the sucrose present it was found necessary to use the Meissl method given in Wein, Zuckertabellen, page 13. The solution used was of such a dilution that the table given on page 183, Bulletin 30, did not apply. To use Meissl's method an extra determination of the direct reducing power of the solution, i. e., before inversion, was rendered necessary. The method is, in short, as follows:

Reagents.—Copper solution: 34.639 grams per 500 cc. Alkaline tartrate solution: 173 grams Rochelle salt and 100 cc. of a sodium hydrate solution containing 516 grams of the base per liter.

Use 25 cc. copper solution, 25 cc. alkaline and tartrate solution, 25 cc. of the inverted solution, and make up to 100 cc. with water. Boil two minutes.

The following results were obtained by these methods:

I. STARCH.

Inversion with HCl.		Märcker method.
Fehling.	Allihn.	
Per cent.	*Per cent.*	*Per cent*
18.143	17.360	16.49
18.234	17.220	16.27
18.102	....	16.52
....	....	16,42
Means: 18.159	17.290	16.428

II. DEXTROSE.

Allihn method.	Fehling gravimetric method.
Per cent.	*Per cent.*
11.38	11.741
11.48	11.707
11.56	11.741
Mean: 11.473	11.729

III. CANE SUGAR.

(*a*) By inversion with yeast.		(*b*) By HCl inversion.
Fehling.	Meissl.	Meissl.
Per cent.	*Per cent.*	*Per cent.*
8.243	11.40	20.43.

IV. GUM ARABIC.

By Tollens-de Chalmot method.
Per cent.
5.931
5.904
5.823
Mean: 5.886

RÉSUMÉ.

Constituents.	Fehling method.	Allihn method.		
	Starch by HCl inversion.	Starch by HCl inversion.	Starch by Märcker method.	Sucrose by HCl inversion.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Starch	18.159	17.290	16.428	16.428
Dextrose	11.729	11.473	11.473	11.473
Sucrose.........................	8.243	11.400	11.400	20.430
Gum arabic	5.886	5.886	5.886	5.886

There is one drawback to the above method, namely, both the difficulty with which the precipitates are washed, the limited washing allowed, so as to not make the resulting filtrates too dilute, as they will not safely bear evaporation, and the difficulty with which such colloidal bodies as gum arabic or dextrin are washed out of precipitates. It seems highly probable, therefore, that most of the above results are too low, especially the gum arabic. The slight reducing power of the dextrine in the last solution had to be neglected, as there was no way of avoiding it. It therefore enters into the result for dextrose, but is so slight as not to materially affect it. The figure for gum arabic seems low. There is a probability that the gum used by the reporter was adulterated, and it would seem important to obtain a figure with the material itself.

The reference committee reported as follows on the recommendations of the reporter on cattle foods:

1. That in crude fiber determinations there be no further division of the classes of substances rich and poor in starch and that one strength of solutions be used for all classes.
2. That the Official method designate 1¼ per cent acid and 1¼ per cent alkali as the strength solutions for crude fiber determinations for all classes of substances and that the 2½ per cent solutions be stricken out.
3. We recommend that the study of methods for separation and determination of the nitrogen-free extract constituents be made the special order for next year's work of the association.
4. That closed digestion and the imitation of animal processes in crude fiber determination be made a point for special study by the next reporter.

The report was approved and adopted by the association.

The following communications from foreign chemists taking part in the work in cattle foods were presented:

RESULTS ON OFFICIAL SAMPLES AND DESCRIPTIONS OF METHODS EMPLOYED.

By Dr. Fassbender.

DETERMINATION OF NITROGEN IN FEEDING STUFFS.

Place 1 gram substance in a Kjeldahl flask of 150 to 180 cc. capacity, add 20 cc. of concentrated sulphuric acid, about 0.7 gram of mercuric oxid and a piece of paraffin the size of a pea. After the acid has thoroughly wetted the substance, warm, at first gently, then more strongly, to strong ebullition, continuing until the contents of the flask become white. Allow to cool, dilute, and wash into a distillation flask. Add 60 cc. of a caustic-soda solution containing potassium sulphid and distill into titrated sulphuric acid. To avoid bumping a little zinc dust is added. The soda solution is prepared by adding 350 grams of potassium sulphid dissolved in 2 liters of water to 11.5 liters of caustic-soda solution of 1.375 sp. gr.

Results obtained on samples sent.

	Nitrogen.	Albuminoids.
	Per cent.	*Per cent.*
Feeding stuff No. 1	0.868	5.43
	.868	5.43
Feeding stuff No. 2	1.600	10.00
	1.550	9.69
Feeding stuff No. 3	7.240	45.25
	7.240	45.25

DETERMINATION OF WATER AND FAT IN FODDERS.

Place 2 grams substance in a weighed porcelain crucible, dry three hours at 100° and weigh. Transfer the dry substance to a capsule made of fat-free filter paper, tightly closed at the bottom, and extract five hours in a Soxhlet extraction apparatus with ether free from alcohol and water. Evaporate off the ether and dry for three hours at 100°. In the case of material containing drying oils, the drying of the substance as well as that of the fat must be done in a current of hydrogen.

	Loss.	Water.	Fat.	Fat.
	Gram.	*Per cent.*	*Gram.*	*Per cent.*
Feeding stuff No. 1	0.1124	5.62	0.0298	1.49
	.1116	5.58	.0294	1.47
Feeding stuff No. 2	.2240	11.20	.0382	1.91
	.2220	11.10	.0394	1.97
Feeding stuff No. 3	.1400	7.00	.2416	12.08
	.1400	7.00	.2410	12.05

ASH IN FEEDING STUFFS.

A platinum crucible is half filled with quartz sand, ignited, cooled in desiccator and weighed. Two grams of substance are placed in this, mixed with the sand, and the crucible ignited. After complete combustion the crucible is cooled and weighed.

	Ash.	Ash.
	Gram.	*Per cent.*
Feeding stuff No. 1	0.1126	5.63
	.1072	5.36
Feeding stuff No. 2	.0360	1.80
	.0364	1.82
Feeding stuff No. 3	.1336	6.68
	.1344	6.72

CELLULOSE IN FEEDING STUFFS.

Place 2 grams of material, from which the fat has been extracted, in a porcelain dish and cover with 50 cc. of 5.5 per cent sulphuric acid and 150 cc. of water. Boil for one-half hour over wire gauze, occasionally adding water to replace that which evaporates. Filter, and wash the residue back into the dish and boil up with water. Once more filter and then wash the residue back into the dish. Add 50 cc. of 5.5 per cent solution of caustic soda and 150 cc. of water. Boil one-half hour over wire gauze, replacing evaporated water from time to time. Filter, return to dish, and boil up with fresh water, repeating this operation a second time. Wash the fiber on the filter several times with hot water, extract with alcohol and ether, and place in the drying bath for some time. Transfer to a platinum crucible. Dry to constant weight, weigh, burn, weigh and deduct the ash from the total obtained.

	Crude fiber.	Crude fiber.
	Gram.	*Per cent.*
Feeding stuff No. 1	0.6879	34.40
	.6981	34.91
Feeding stuff No. 2	.0441	2.21
	.0510	2.55
Feeding stuff No. 3	.0920	4.60
	.0970	4.85

THE DETERMINATION OF FIBER IN SCOTLAND—OFFICIAL SAMPLES.

By A. P. Aitken.

H. W. Wiley:

Dear Sir: Great pressure of work has prevented my overtaking more than the crude-fiber section of the analytical work proposed in your letter of 25th April. The samples only arrived on 21st May.

Annexed is the record of 24 analyses in 8 sets of 3 each.

Sets Nos. 1. 2, and 3 were done in exact accordance with the instructions sent me. The others were done by various modifications of my own method, with the object of finding which modification brought out results most nearly resembling the American Official methods.

I am accustomed to using method No. 8 in analyzing feeding stuffs.

The apparatus I use is simpler and has some advantages over that described by you.

The material (usually 1 gram) is put into a beaker of about 150 cc. capacity and 100 cc. of the acid or alkali is added; the beaker is then covered with a watch glass and let into a steam bath, along with other five, it may be. The bath is simply a long rectangular box of copper with a movable lid. Water is admitted to a gutter about 1¼ inches deep in the bottom of the bath, and it is supplied at a constant level from a cistern attached to it.

The water in the gutter is kept boiling by means of a row of gas jets. The box is therefore full of steam and the contents of the beakers at 100° C., but not boiled. There is therefore no frothing, and the beakers need no attention whatever. Any advantage that may accrue from boiling may be attained by prolonging the time during which the beakers are immersed in the bath. Beakers are otherwise more convenient than flasks; the material is easily washed out of them. I find also that the physical condition of the fiber exposed in that way is such as to permit of its being filtered and washed more rapidly than after boiling.

My assistant, Mr. George Burns, undertook the practical work of these analyses, and I can guaranty their accuracy.

Woody-fiber determinations, by A. P. Aitken.

	Samples.		
	I.	II.	III.
1. American Official method:	*Per ct.*	*Per ct.*	*Per ct.*
No. 1, 1¼ per cent H_2SO_4 one-half hour; 1¼ per cent NaOH one-half hour.	34.36	2.46	4.11
2. No. 2, 2½ per cent H_2SO_4 one-half hour; 2½ per cent NaOH one-half hour.	30.90	2.04	3.55
3. American method, No. 3, 1 per cent HCl two hours; 1 per cent Na_2CO_3 two hours	35.95	3.10	7.40
4. Own method, modified:			
1¼ per cent H_2SO_4 one-half hour; 1¼ per cent NaOH one-half hour	35.45	2.65	4.65
5. 2½ per cent H_2SO_4 one-half hour; 2½ per cent NaOH one-half hour	31.10	2.30	4.10
6. 2½ per cent H_2SO_4 one-half hour; 1¼ per cent NaOH one hour	31.60	2.70	4.20
7. 2½ per cent H_2SO_4 one hour; 1¼ per cent NaOH two hours	27.70	2.15	3.60
8. Own method, 5 per cent H_2SO_4 one hour; 2½ per cent NaOH two hours	26.32	2.10	3.25

No starch was found in any of the crude fibers, and only in the case of No. 3 (I and III) was there any albuminoid matter, but the quantity was too small to be regarded, viz, 0.03 to 0.05 grams nitrogen.

No further papers offering on cattle foods, the President named as committee on revision of statements of methods of analysis, to report next year, Messrs. Van Slyke, de Roode, and Bigelow.

The next business was the reading of the report on dairy products, which, by agreement, was postponed until the following morning.

Mr. Kilgore offered an amendment to the constitution, suggested by Mr. Battle, which was read by the Secretary, as follows:

> No changes shall be made in the methods of fertilizer analyses until an opportunity shall have been given to all institutions exercising official fertilizer control to test the proposed changes.

This, after discussion, was referred by the President to a committee, consisting of Messrs. Wiley, Ross, and Winton, for report.

The association then adjourned until the next morning.

THIRD DAY.

SATURDAY—MORNING SESSION.

The President called the association to order at 9:30 a. m. Reports of committees were called for.

Mr. WHEELER. I think Dr. Peter had the record of the committee, but the report of the committee of five is on the work by Mr. Patterson and on two matters referred to the committee yesterday. In the absence of Dr. Peter we will report on the two matters referred especially, and Mr. Patterson has the matter pertaining to his recommendations.

In the matter of striking out the Ruffle method and in regard to the Tiemann-Schultze process, the committee reported as follows:

> The committee of five recommended that the Ruffle method be retained as one of the official methods. The committee also recommended that the Tiemann-Schultze method for the determination of nitrogen in nitrates be adopted as a provisional method and not as an official one, until the method has been tested by the association.

Adopted.

Mr. PATTERSON. In the matter of cattle foods, the only recommendation made was that in section 2, page 191, in feeding stuffs rich in starch, the association strike out the use of the 2.5 per cent solution, leaving only one strength to be used. This was equivalent to striking out the whole section. It was also suggested that the reporter be instructed to further investigate the subject of the imitation of the processes of animal digestion in determining crude fiber.

Mr. HUSTON asked, if the whole of section 2 were stricken out, whether it would not also be well to strike out the portion in italics of section 1, page 190, "in feeding stuffs poor in starch?"

Mr. LINDSEY objected, on the ground that he did not like so much haste in changes of methods.

Mr. WOLL. The reporter of last year had this subject under consideration, and came to the same conclusion, that there was no ground for division of the methods in case of the poor in starch and rich in starch. No work has been done by the association that shows that there is any real cause for a separation of the methods.

The recommendation was adopted.

An amendment was offered by Mr. Frear, including Mr. Huston's suggestion, to strike out the words in italics in section 1. Adopted.

The SECRETARY. The committee on amendment to the constitution is ready to report. The committee has considered the recommendation by Mr. Battle, and recommends its adoption with an amendment inserting except by "unanimous consent" after "analyses." The committee recommends the adoption of this amendment.

The committee is of the opinion that all radical changes in methods should go for at least one year to the institutions interested in them before official adoption by the association; and by inserting this little amendment it is hoped the clause in the constitution will be flexible enough to permit unimportant changes, while it will prevent radical changes until due opportunity has been had for their proper consideration.

Moved and seconded that the report of the committee be adopted. Carried.

The PRESIDENT. The question now is upon the adoption of the amendment. I will read the section of the constitution in reference to this point:

> Only such chemists as are connected with institutions exercising official fertilizer control shall vote on question involving methods of analyzing fertilizers.

Mr. VAN SLYKE. This amendment would practically include only official chemists.

Mr. McDONNELL. I think I would rather see this amendment say "official chemists" than "institutions." They are the ones to act in the matter.

After some further discussion Mr. Van Slyke moved that the amendment be amended so as to read "official chemists having charge of fertilizer work" instead of "institutions."

This was adopted.

The PRESIDENT. The question now is upon the adoption of the amendment to the constitution. Will the secretary read the amendment as amended?

The SECRETARY:

> No changes shall be made in the methods of fertilizer analyses except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

Mr. PATTERSON wished to know if, after the opportunity to consider had been given, the usual vote could decide to make a change. He thought if the amendment was not flexible enough to allow that, no change could be made in the fertilizer analyses.

The PRESIDENT. After the opportunity has been given, as I understand it, the regular method of procedure is in order. Before a change can be made it has to be submitted to these various chemists.

Mr. PATTERSON. Then before you can even change the number of cubic centimeters, you have to refer it back to these institutions. The rule seems ironclad.

Mr. WHEELER pointed out that the change made by the committee provides for all Mr. Patterson objected to. It allows unimportant changes to be made, provided there is no objection.

Mr. LINDSEY did not believe in the amendment at all. He thought the association was working all right as it was.

The question being called for, the secretary ascertained that there were fourteen official chemists present entitled to vote. Nine voted for, and four against the amendment. It was pointed out that nine to fourteen is not a two-thirds vote, which the President remedied by stating that he would vote for the amendment.

By this vote it was carried.

Mr. KILGORE moved, in reference to the vote of yesterday on phosphoric acid, that on page 170, second line, second paragraph, total phosphoric acid, that 2.5 per cent ammonia be used, so as to be uniform.

Carried.

The final changes in potash methods, adopted by the association, are as follows:

CHANGES IN METHODS FOR POTASH DETERMINATION, ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, AUGUST 24, 1894.

On page 178 of the proceedings for 1893, in the fifth line from the bottom, omit "(4)" and insert "Method of solution for" immediately preceding the word "Organic." On the same page, in the last line from the bottom, omit "(1)" and add the following: "The Lindo-Gladding or Alternate Method;" also transfer the last paragraph on page 178 to page 179, immediately preceding "factors," and change the heading from a subheading to a general one. On page 179, in the first line from the top, after the words "boil for," insert "30" in place of "10," and insert "300" in place of "200." On the same page, in the third line from the top, insert after "sample has" the word "over," and omit in the same line "to 15," and also "(kainit)." In the fourth line of the same page omit "from 2 to 3," and insert in its place "less than 10."

The following line of investigation was suggested to the reporter for 1895, viz: Further study of the double decomposition resulting in the treatment of K_2PtCl_6 with the ammonium chlorid wash solution which is used in the Lindo-Gladding method.

Also further comparative tests of methods in the presence of maximum quantities of impurities in form of sulphates.

The President called for the report on dairy products.

Mr. FARRINGTON read the report, and also reports received from Messrs. Winton, Spencer, and Van Slyke too late to be used in his work of this year.

REPORT ON DAIRY PRODUCTS.

The 1894 reporter on dairy products of the Association of Official Agricultural Chemists does not include in his report an abstract of the chemical dairy literature of the past year, as it is available to all the chemists of the association and can be read by them in the original publications as soon as new ideas appear in the various journals. It is also well known that the editor of the department of dairying attempts to give abstracts of current literature in the Experiment Station Record, which publication is more or less read by all the chemists of this association.

The first report on dairy products made by this association was at the fourth annual convention in 1887, by H. W. Wiley. Four samples were sent out by him to members of the association for analysis. These samples were: No. 1, oleomargarin; No. 2, butterin; No. 4, butter; No. 4, Philadelphia print butter. Results of analyses of these samples were received from eight analysts. A summary of these results is given in the following table, although not everyone of these determinations was made by each analyst. The extreme and average results obtained by the eight analysts on each sample are given:

	Specific gravity.	Melting point.	Vol. acids cc. $\frac{N}{10}$ alk.	Soluble acids.	Insoluble acids.	Saponif. equivalent.	Water.	Salt.	Curd.
Oleomargarin:		C°.	cc.	*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*
Maximum	.9079	33.6	0.6	0.22	95.25	287.2	7.72	2.18	1.48
Minimum	.9050	31.8	.31	.21	91.26	275.8	6.25	1.82	.34
Average	.9063	32.4	.46	.215	94.01	281.6	6.80	2.05	.83
Butterin:									
Maximum	.9071	41.5	.90	1.19	96.13	288.7	8.06	2.21	2.34
Minimum	.9038	33.3	.29	.10	93.40	263	6.60	1.88	.27
Average	.9050	38.6	.48	.61	94.80	282.1	7.15	2.07	1.12
Butter:									
Maximum	.9121	36	13.43	5.56	90.09	250.4	11.06	5.11	3.70
Minimum	.9086	28.3	12.30	4.48	84.29	241.2	8.97	3.86	.41
Average	.9095	33.4	12.83	5	87.21	246.4	10.22	4.58	1.73
Print butter:									
Maximum	.9137	35.4	15.90	5.81	88.60	250	11.36	1	1.79
Minimum	.9105	26.6	14.31	5.73	83.37	230.8	8.21	.79	.64
Average	.9115	32.3	15.06	5.76	86.68	244.5	10.09	.86	1.15

The fifth annual convention, 1888, H. W. Wiley, reporter. No samples were sent to members for analysis The report consists of "a brief résumé of the work which has been done during the year."

The sixth annual convention, 1889, H. W. Wiley, reporter. Three samples were sent out for analysis. They were: No. 1, oleomargarin; No. 2, butterin; No. 3, butter. Detailed instructions for determining the melting point, specific gravity, and volatile fatty acids were sent with the samples. Reports of analyses were received from 22 analysts, some of them giving minute details of weighings, preparation of solutions, reagents, etc. Leaving out the results of six analysts on specific gravity, seven on melting point, and eight on volatile fatty acids, the average and extreme results obtained by the 22 analysts on these 3 samples were as follows.

	Specific gravity.			Melting point. C°.			Volatile acids cc. $\frac{N}{10}$ alk.		
	Oleomargarin.	Butterin.	Butter.	Oleomargarin.	Butterin.	Butter.	Oleomargarin.	Butterin.	Butter.
Maximum	0.9009	0.8986	0.9030	31.4	36.6	34.50	1.48	2.55	29.58
Minimum	.8953	.8949	.8991	27.8	33.2	32	.42	1.11	25.34
Average	.8972	.8967	.9006	29.6	35.17	33.64	.87	1.71	27.65

The seventh annual convention, 1890, E. H. Jenkins, reporter. The reporter sent out one sample of creamery butter with instructions for the determination of water, casein, fat, and ash (salt). Eleven analysts sent in reports of analysis of this sample. The present official method was used in obtaining these results.

	Water.	Casein.	Fat.	Ash.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Maximum	13.02	1.95	81.90	5.11
Minimum	12.64	.88	80.02	4.58
Average	12.79	1.31	80.99	4.91

The eighth annual convention, W. W. Cooke, reporter. A sample of sweet milk was sent to each of 10 analysts. The following results were reported:

	Solids.	Fat.	Casein.	Sugar.	Ash.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Maximum	13.19	4.07	3.69	5.27	0.84
Minimum	12.91	3.80	3.12	4.70	.72
Average	13.05	3.91	3.45	4.92	.78

These results were obtained by the use of several methods. The solids by drying the milk at 100° and 105° C., on sand and on asbestos; the fat by weighing the ether extract from the milk dried on paper, sand, and asbestos, and also by the Beimling, Parsons, and Babcock volumetric methods.

One sample of sour buttermilk was also sent out by the reporter to six analysts. This sour milk was neutralized with ammonia in some cases, and the fat determined both gravimetrically and by the volumetric methods of measuring the fat. A summary of the results is as follows:

	Solids.	Fat.
Maximum	9.97	0.65
Minimum	9.07	.20
Sweet sample	9.98	.69

The ninth annual convention, S. M. Babcock, reporter—The members of the association were requested to investigate the following points:

1. Does the saponification of fats by means of glycerol and caustic soda as proposed by Leffman and Beam give identical results with the present official method?
2. Are the results with a block-tin and glass condenser the same?
3. Is it necessary to melt the free fatty acids before distillation?
4. Methods of cheese analysis.

No samples were sent out. Results of investigations were received from five analysts. These results are briefly summarized as follows: O'Brien's results are the average of five trials. Penny made twenty-six determinations, all on the same sample of butter, including eleven estimations of volatile acids by the official method and fifteen by Leffman and Beam's method. A glass condenser was used in eleven determinations and a block-tin condenser in fifteen determinations; also twenty-two estimations of volatile acids with previous melting of fatty acids, and four estimations without previous melting of fatty acids.

Heilman's results were from three samples of butter, Bartlett's from six butters, and Babcock's from four samples, making a total of thirteen samples, on which a comparison was made of the Leffman and Beam with the official method of saponifying fats. The average of these results expressed in cubic centimeters, $\frac{N}{10}$ alkali, is as follows:

Analyst.	Method.		Condenser.	
	Official.	Leffman and Beam.	Glass.	Block tin.
O'Brien	26.01	24.01	....	....
Penny	28.69	28.72	28.74	28.68
Heilman	26.01	25.35	....	....
Bartlett	29.40	29.25	....	....
Babcock	29.25	27.82	28.72	28.34
Average	27.87	27.03	28.73	28.51

Dr. Babcock was convinced "that the volatile fatty acids act upon the tin condensers and are partially neutralized. In regard to the necessity of melting the fatty acids before distillation the work is not conclusive, as only two trials have been reported."

The work on cheese analysis included the estimation of water by drying over sulphuric acid and at 100° C.; also the estimation of fat in these dried residues. The average results by the different analysts are grouped together in the following table; they include the analyses of 28 samples of cheese:

Analyst.	Water.		Fat.			
			Direct extraction of residue.		Short's method.	Babcock's method.
	Dried over H_2SO_4	Dried at 100° C.	Dried over H_2SO_4	Dried at 100° C.		
McElroy	36.07	37.15	32.50		33.42	
Winton	35.67	36.89				
Lyon	36.74	36.93	33.95		34.43	
Snyder	32.86	33.96	37.62		37.50	
Babcock	27.43	28.73	39.39	38.34	39.72	39.49
Average	33.75	34.73	35.86		36.27	

The tenth annual convention, A. L. Winton, reporter.—The special subjects investigated were—

1. Drying of cheese and ether extract in air and in hydrogen.
2. Determination of solids and fat in milk by drying with asbestos in a perforated metal cylinder.
3. Calculation of solids in milk from its specific gravity and per cent fat.

No samples were sent out by the reporter. Results of investigations were received from ten analysts.

1. *Drying of cheese and ether extract in air and in hydrogen.*—The results obtained by drying cheese and ether extract at 100° C. in air as compared with drying in hydrogen, show that about the same loss in weight was obtained by both methods of drying, although in some samples there was a difference of about 0.5 per cent, and in one sample of 2 per cent. These were the results obtained on a total of 9 samples of cheese as reported by four analysts and 11 analyses of ether extract reported by six analysts.

It was clearly shown that when cheese is dried in both air and hydrogen there is a gradual loss in weight even when some samples were heated forty hours; the loss in weight continued to be noticed, although the greater part of the loss takes place in the first four hours of heating.

The reporter recommended that the determination of crude protein in cheese should be made by subtracting the sum of the water, ash, and fat from 100 instead of multiplying the nitrogen by a factor. This recommendation was made because of the variety of nitrogenous substances in cheese and the consequent inaccuracy of any one factor for them all.

2. *Determination of solids and fat in milk by drying with asbestos in a perforated metal cylinder.*—Results are given of analyses of about 20 samples of milk by eight analysts. The total solids and fat were determined in the milk samples by this as well as other asbestos methods, the paper coil and sand methods, milk dried alone and in hydrogen. Excepting the per cent of total solids obtained in milk by drying in hydrogen, which result was 0.15 per cent lower than that obtained by drying in air, there was less than 0.1 per cent difference between the per cent of fat and solids obtained by drying milk with asbestos in a perforated metal cylinder and those obtained by the other methods of drying and extracting milk. The reporter also

states that "for simplicity and convenience the cylinder method can hardly be equaled."

Determinations of fat in milk by weighing the ether extract obtained from solids dried in air as compared with drying the solids in hydrogen.—Analyses of 12 samples by three analysts, who made this comparison on each sample, show that "the results by the two methods are practically the same."

3. *Calculations of the total solids in milk from the per cent of fat and the specific gravity.*—These calculations were made by the use of three different formulas, that of Fleischmann, Hehner and Richmond, and Babcock.

A comparison of the average gravimetric with the average calculated results on solids in whole milk by each of the formulas show that in 39 samples of milk, as reported by six analysts, the closest agreement was obtained by using the formula of Hehner and Richmond when solids were determined by drying 34 samples with sand or asbestos, but by Fleischmann's formula when 4 samples of milk were dried alone for determination of solids.

In addition to the reports made each year on dairy products, the following papers have been read and printed in the proceedings of the Association:

The fourth report, page 23, "Average of analyses of American butters," by Edgar Richards; page 25, "Variations in composition of butter," by S. M. Babcock.

The fifth report, page 24, "The milk of Washington not normal," by C. Richardson; page 25, "Notes on the method for soluble and insoluble fat acids;" page 36, "Refractive index of butter fat," by H. W. Wiley.

The eighth report, page 22, "Analyses of sour milk," by W. W. Cooke; page 26, "Composite milk samples in the laboratory," by G. E. Patrick.

The tenth report, page 109, "The determination of casein in cow's milk;" page 116, "The determination of fat in cheese;" page 117, "The determination of acidity in milk," by L. L. Van Slyke; page 119, "The determination of solids and fat in milk by drying with asbestos in a perforated metal cylinder," by A. L. Winton.

The reporter on dairy products this year sent out the following circular letter to fifty chemists:

CHAMPAIGN, ILL., *March 7, 1894.*

DEAR SIR: The 1894 reporter on dairy products of the Association of Official Agricultural Chemists makes the following suggestions in regard to an investigation on milk:

In a sample of sweet milk determine—

(1) The specific gravity by weight and also by lactometer, if one is available, noting the temperature of the milk at each observation.

(2) The per cent of total solids by weight, stating temperature of oven and length of time heated, the number of times weighed after heating began, and what kind and amount of material was mixed with the milk when heated.

(3) The per cent of fat by extraction of dried solids with ether or any other satisfactory solvent, and weight of the fat so obtained, stating solvent used, length of time the material was extracted, and also temperature and time of drying fat.

State the exact or approximate age of each sample of milk analyzed, and, if possible, determine the specific gravity of one or more samples of milk within one, two, three, and four or more hours after such sample is drawn from the cow, noting the change in specific gravity of the sample during this time. A determination of the total solids and fat is also desired in the milk whose specific gravity is observed at intervals of one or more hours.

From the specific gravity and per cent of fat found in the samples calculate the total solids or solids not fat by each of the three formulas given on page 107 of the Proceedings of the Tenth Annual Convention of the Association of Official Agricultural Chemists. It is hoped that these observations may be made on a great variety of pure milks, and the results sent to the undersigned as soon as possible.

Very truly, yours,

E. H. FARRINGTON.

The following observations on the change in specific gravity of sweet milk were made by the writer. The different samples of milk were received at the laboratory from one to three hours after the milk was drawn from the cows. The specific gravity was determined by means of a pyknometer. This determination was repeated every few hours on each sample of milk in order to note any changes in the specific gravity. The results are all calculated to 15° C. and are expressed in the usual way of reading a lactometer, such as 1.0357 is 35.7, etc.:

	Cow No. 1.	Cow No. 2.	Cow No. 3.	Cow No. 4.	Cow No. 5.	Herd milk.
Age of milk, hours:						
1	35.7	36.0	....	....	....	....
2	36.0	....	32.90	33.1	34.9	34.00
3	35.9	....	....	....	....	....
4	35.9	36.3	....	....	....	....
6	35.9	36.7	....	....	....	....
10	36.0	36.7	....	....	....	34.00
26	....	....	32.80	33.4	36.0	34.00
34	....	....	33.00	32.9	35.5	....
Fat, per cent by weight	3.8	4.2	2.85	2.9	4.3	3.40
Solids, per cent by weight	13.3	14.0	11.25	11.6	13.9	12.65

This limited number of observations on this point indicate that the change in specific gravity of sweet milk is not very great, and that this change was not uniform in all these samples. In five other samples no difference was observed in the specific gravity as indicated by a lactometer when the samples were three or twelve hours old. A comparison of the calculated and weighed amount of solids was also made by the writer in 15 samples of milk.

Each sample of milk was weighed on sand and also on asbestos in a perforated metal cylinder. After drying about four to six hours in a bath surrounded by boiling water, until there was no further loss of weight, the solids were weighed, and the percentages thus found are given in the table. The dried solids on both sand and asbestos were then extracted with ether in a Soxhlet extractor for four hours. The fat thus obtained was dried in a water oven and weighed. The percentages of fat so obtained are recorded in the table. The fat in each sample was also estimated by the Babcock method, and a lactometer reading of each sample was also observed. The per cents of solids found by drying and weighing on sand and on asbestos show a very close agreement, as do also the per cents of fat obtained by ether extraction of the solids dried on sand and on asbestos and by the Babcock method. The per cent of solids in each milk was also calculated from the lactometer reading and the per cent of fat, using each of the four following formulas for these calculations. In all these calculations the writer has used the per cent of fat obtained by the Babcock method, as it was practically identical, from a working chemist's standpoint, to the per cent of fat found by weighing the ether extract.

Fleischmann:[1] $t=1.2f+2.665\,\frac{100S-100}{S}$

Babcock:[2] Solids not fat $=\left(\frac{100S-Sf}{100-1.0753Sf}-1\right)\times(100-f)2.6$

Hehner and Richmond:[3] $t=\frac{f+.2182S}{.859}$ If $\frac{S}{t}>2.5$

Correct the value obtained for t by adding $.05\left(\frac{S}{t}-2.5\right)$

Richmond:[4] $t=2.625\,\frac{100S-100}{S}+1.2F$

The following table gives a summary of these results:

Milk.	Lactometer at 15° C.	Per cent of fat.			Per cent of total solids.					
		Ether extraction of—		Babcock method.	Weighed on—		Calculated by formula of—			
		Sand.	Asbestos.		Sand.	Asbestos.	Babcock.	Fleischmann.	Hehner and Richmond.	Richmond.
One cow	35.0	3.83	3.86	3.8	13.38	13.21	13.71	13.56	13.31	13.43
Do	36.0	4.27	....	4.2	14.06	13.95	14.46	14.30	14.02	14.16
Do	34.0	3.48	3.47	3.4	12.60	12.70	12.98	12.84	12.70	12.73
Do	33.0	2.77	2.85	2.9	11.30	11.18	12.10	11.99	11.76	11.86
Do	33.0	2.92	2.86	2.9	11.62	11.58	12.10	11.99	11.76	11.86
Do	35.0	4.25	4.34	4.3	13.97	13.85	14.32	14.16	13.90	14.03
Herd	32.8	....	4.33	4.4	13.53	13.45	13.90	13.73	13.45	13.61
Do	33.5	4.77	4.84	4.8	14.00	13.94	14.54	14.39	14.09	14.26
One cow	33.0	5.36	5.49	5.5	14.66	14.58	15.26	15.11	14.78	14.98
Skim	38.6	.96	.94	1.0	10.72	10.75	11.22	11.08	11.02	10.95
One cow	33.6	....	5.79	5.8	15.05	....	15.76	15.62	15.30	15.49
Do	30.6	6.93	7.05	7.0	15.43	....	16.41	16.31	15.92	16.20
Do	30.6	4.18	....	4.1	12.31	....	12.90	12.83	12.54	12.71
Do	28.5	4.00	4.01	4.2	11.48	....	12.49	12.43	12.13	12.31
Do	33.6	....	....	5.0	14.42	....	14.78	14.66	14.35	14.53

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[1] J. Ldw. 1883, 251.

[2] Wis. Exp. Sta. Rep. 1891, 292.

[3] Analyst, **13**, 26.

[4] See his letter in this report.

These results, like those of similar work reported to this convention by Winton last year, show that the formula of Hehner and Richmond gives results that compare the nearest to the per cent of solids obtained gravimetrically.

Nearly all the results obtained by using a formula for calculating the total solids are higher than the dried and weighed solids. Babcock's formula gives the highest figures, Fleischmann's the next highest, Richmond's next, and that of Hehner and Richmond, with a few exceptions, very nearly the same per cent of solids as was found gravimetrically.

This is also the case in the following work, sent by J. W. Fields, of the Pennsylvania Experiment Station:

Sample milked at 6:30 a. m. Samples weighed out for solids and fat at 10 a. m.

Specific gravity by weight at 15° C.:

At 10 a. m 1.0336

At 1 p. m 1.0335

Total solids by weight (after drying 10 hours) .... $\left\{\begin{matrix}\text{13.29 per cent.}\\ \text{13.23 per cent.}\end{matrix}\right.$

Fat: By weight $\left\{\begin{matrix}4.13.\\ 4.14.\end{matrix}\right\}$ By difference $\left\{\begin{matrix}4.11\\ 4.11\end{matrix}\right\}$ after extracting with ether purified over lime and distilled, then over metallic sodium and distilled.

Total solids calculated from fat and specific gravity:

Fleischmann 13.58

Babcock 13.69

H. and R. 13.29

The samples were dried and extracted in glass tubes containing about 4 grams short asbestos. The end of the tube was covered with two layers of linen, with one of filter paper between.

The following report was received from H. Droop Richmond, of England. He has used the four formulas for calculating the solids not fat in 75 samples of milk, and, as the table shows, 50 per cent of his results are nearest to the gravimetric figures when he used Richmond's formula, 24 per cent with each of Fleischmann's and Hehner and Richmond's, and about 2 per cent with Babcock's formula:

50 CLANRICARDE GARDENS, BAYSWATER, W.,
April 30, 1894.

MY DEAR SIR: I send herewith a selection of results for comparison with those of American chemists. They are not all done specially for this work, but as they were done with a similar object and are as accurate as possible, I think that you may find it of interest to compare them with the others you will receive. I propose to devote the time at my disposal for this work to determining the change in specific gravity in milk after milking. As my laboratory is stationed in London, away from the farms, I shall have some difficulty in procuring many samples, and I think that 25 to 30 will be the most I can do.

I have ventured to include a new formula, $T=2.625\frac{100S-100}{S}+1.2F$, in my results, as I believe it is more accurate, and I send my results off soon, as I think you may like to compare this also.

I see you allow considerable latitude as to the methods to be used, and I trust that you will find my methods sufficiently in accord with the official methods of the Association of Official Agricultural Chemists to be comparable.

Yours, very sincerely,

H. DROOP RICHMOND.

E. H. FARRINGTON,
Champaign, Ill., U. S. A.

METHODS OF ANALYSIS.

Specific gravity.—All figures in Table I were obtained by weighing 10.113 cc. (i. e. a volume holding 10.113 grams water at 15.5° in air) of milk at a temperature of 15.5°, measured by a thermometer which had been standardized at the Royal Observatory at Kew; temperature accurate to 0.1°.

Total solids.—About 3 grams of asbestos were placed in a platinum basin, ignited, and weighed; 5 grams (approximated) of milk were measured in a pipette and allowed to run into the asbestos, and again weighed; the drying was done for three hours on a steam bath (temperature about 96° to 98°), and then till constant in an air bath (temperature 102°), the time being eighteen to twenty-four hours; two weighings were always performed, and if the second (about four to five hours after the first) differed more than a milligram, which occurred in two cases, a third was made after about three hours more; in two cases (Nos. 61 and 62) the difference between duplicates was more than 0.04 per cent. (Mean error, $\pm$ 0.015.)

Fat.—Five grams of milk were run from the same pipette on to Schleicher and Schüll's extracted coils, care being taken that the milk was delivered in the same time and in the same manner as for the total solids; the mean weight of milk taken for the total solids was assumed to be the weight taken for the fat; in no case did the weights of milk taken for total solids differ by sufficient to cause an error in the second place of decimals in the fat. The coils were first air-dried and then placed in the air bath for one hour; the flasks for the extraction were tared against an empty flask in which a blank extraction of a coil was performed; the extraction was performed in Soxhlet extractors for four hours, with ether (washed with water and dried for a week over an excess of calcium chloride, and distilled). The fat was dried in the flasks for one-half hour in the same air bath, air being blown in and the flask rotated every ten minutes. Extreme difference between duplicates, 0.05 per cent. (Mean error, $\pm$ 0.027.)

All milks were about twenty-four hours old when analysed, and were quite sweet (acidity calculated as lactic acid less than 0.22 per cent).

The balance used was a long beam Oertling, sensible to 0.1 milligram; the weights were by Sartorius and had been carefully standardized; no weight was more than 0.3 milligrams out. Weighings were made in air, and corrections were not made of any kind.

In these milks lactometer readings were not taken, the gravimetric determinations, however, never differed in duplicates by more than 0.0001.

A large number of these results were used to calculate a new formula, $T=2.625\frac{100\ S-100}{S}+1.2F$, and I have ventured to include the results calculated by this formula, especially as I find that in the results (average of all observers) given in the report on dairy products last year it agrees better than any of the others. In Table I the formula which agrees best is indicated by a line in pencil drawn under the result by that formula; where two agree equally well, a cross line is added and this is counted as a half result.

FORMULAS.

Of the four formulas I have used, two only, those of Fleischmann and myself, are correct in principle; these two assume that the specific gravity is a direct function of the percentage by weight (i. e., grams per 100 cc.) of both fat and solids not fat; this is manifestly true for the fat, as it is not in solution, and I have made the following determinations to show that it holds for the solids not fat within the limits of experimental error. A poor skim milk was used (fat=0.26).

Percentage.	T. S. =T. Sp. g. =S.	$\frac{100\,S - 100}{S} \div T$
9.280	1.03544	0.3688
8.758	1.03343	0.3693
8.318	1.03170	0.3694
7.777	1.02950	0.3684
7.456	1.02829	0.3690
6.455	1.02439	0.3688

In the formula of Hehner and myself it is assumed that the specific gravity is a direct function of percentages by weight of fat and solids not fat; the correction has no scientific meaning, and expresses only experimental error.

In Babcock's formula the specific gravity is a direct function of the percentage by volume of fat and serum, but a direct function of the percentage by weight of the solids not fat in the serum; this accounts for its cumbrous form.

In the formulæ of Babcock, Fleischmann, and myself it was assumed that the specific gravity of fat was 0.93 and that of the solids not fat found experimentally.

Hehner and I made each pair of our results into simultaneous equations, and obtained $\frac{4\,4}{2}$. constants for the formula, and took the numerical average as being correct; both our methods and mode of calculation were not free from error, and it is due to a compensation of errors that the results come out as well as they do

TABLE I.—*Comparison of determinations of specific gravity with those obtained by lactometers.*

Sp. gr. (gravimetric).	Lactometer reading.	Temperature.	Sp. gr. at 60° F.	Lactometer.	Sp. gr. (gravimetric).	Lactometer reading.	Temperature.	Sp. gr. at 60° F.	Lactometer.
		° *F.*					° *F.*		
1.0336	33.8	60	1.0338	A		33.0	64	1.0336	D
1.0337	34.0	60	1.0340	A	1.0327	32.7	60	1.0327	A
1.0353	35.4	60	1.0354	A		32.5	58	1.0322	B
1.0353	35.5	60	1.0355	A		32.7	61	1.0329	C
1.0356	35.6	60	1.0356	A		32.5	61	1.0327	D
1.0357	36.0	56	1.0355	A		33.0	55	1.0324	E
	35.8	58	1.0355	B	1.0326	32.6	60	1.0326	D
	36.3	55	1.0357	C	1.0325	33.0	57	1.0326	D
	36.0	60	1.0360	D	1.0324	33.0	55	1.0324	D
1.0335	32.8	64½	1.0335	A	1.0315	31.5	60	1.0315	D
	33.0	64	1.0336	B	1.0324	32.6	58	1.0323	D
	32.8	64½	1.0335	C	1.0323	32.8	55	1.0322	D

I have not deemed it necessary to multiply these results, as they only show the error that may be expected from the use of lactometers.

TABLE II.—*Analyses of milk.*

Sp. gr. at 15.5°.	Total solids.	Fat.	Solids not fat.	Solids not fat by—			
				Babcock.	Fleischmann.	Hehner and Richmond.	Richmond.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1.0614	20.84	4.73	16.11	16.82	16.37	16.34	*16.25*
1.0360	11.68	2.06	9.62	9.80	*9.68*	9.49	9.54
1.0333	12.82	3.59	9.23	9.43	9.30	9.04	*9.17*
1.0333	12.84	3.64	9.20	9.44	9.31	9.05	*9.18*
1.0333	12.88	3.70	9.18	9.50	9.37	9.11	*9.24*
1.0330	12.50	3.32	9.18	9.28	*9.18*	8.93	9.05
1.0329	12.48	3.33	9.15	9.26	*9.16*	8.91	9.03
1.0329	11.97	2.85	9.12	*9.16*	9.06	8.82	8.93
1.0318	13.74	4.64	9.10	9.26	*9.12*	8.84	9.00
1.0326	13.04	3.99	9.08	9.33	9.23	8.97	*9.10*
1.0323	12.96	3.92	9.04	9.23	9.12	8.84	*8.99*
1.0320	13.09	4.06	9.03	9.18	*9.08*	8.79	8.96
1.0325	12.75	3.72	9.03	9.24	9.14	8.86	*9.01*
1.0321	12.80	3.78	9.02	9.15	*9.04*	8.78	8.91
1.0322	12.80	3.79	9.01	9.18	9.08	8.82	*8.95*
1.0317	13.04	4.05	8.99	9.10	*8.96*	8.72	8.83
1.0321	12.39	3.40	8.99	9.07	*8.97*	8.71	8.85
1.0349	9.27	.28	8.99	9.13	9.04	*8.97*	8.91
1.0320	12.83	3.85	8.98	9.14	*9.03*	8.76	8.91
1.0320	12.42	3.44	8.98	9.05	*8.94*	8.69	8.82
1.0322	12.76	3.79	8.97	9.18	9.08	8.82	*8.95*
1.0317	13.09	4.13	8.96	9.12	9.02	8.74	*8.90*
1.0318	12.89	3.93	8.96	9.10	*8.98*	8.72	8.86
1.0328	12.44	3.48	8.96	9.27	9.16	*8.90*	9.03
1.0327	12.42	3.46	8.96	9.24	9.13	8.87	*9.00*
1.0328	12.62	3.67	8.95	9.32	9.19	*8.93*	9.06
1.0321	12.60	3.65	8.95	9.13	*9.01*	8.75	*8.89*
1.0332	11.86	2.92	8.94	9.25	9.14	*8.90*	9.01
1.0324	12.59	3.64	8.95	9.20	9.09	8.83	*8.96*
1.0319	12.85	3.92	8.93	9.15	9.02	8.75	*8.89*
1.0324	12.58	3.65	8.93	9.20	9.10	8.82	*8.97*
1.0322	12.42	3.49	8.93	9.11	9.01	8.75	*8.89*
1.0321	12.14	3.21	8.93	9.03	*8.92*	8.67	8.80
1.0320	12.69	3.77	8.92	9.12	9.02	8.74	*8.90*
1.0320	12.67	3.75	8.92	9.12	9.02	8.74	*8.90*
1.0323	12.52	3.60	8.92	9.17	9.04	8.79	*8.91*
1.0322	12.56	3.65	8.91	9.15	9.04	8.78	*8.91*
1.0327	12.51	3.61	8.90	9.27	9.16	*8.89*	9.03
1.0326	12.41	3.51	8.90	9.22	9.11	*8.85*	8.98
1.0327	11.91	3.01	8.90	9.14	9.04	8.79	*8.91*
1.0319	12.61	3.73	8.88	9.09	8.98	8.71	*8.86*
1.0326	12.58	3.70	8.88	9.27	9.15	*8.88*	9.02
1.0326	12.29	3.41	8.88	9.20	9.09	*8.82*	8.96
1.0322	12.39	3.53	8.86	9.11	*9.02*	8.76	*8.90*
1.0325	12.32	3.46	8.86	9.18	9.09	*8.81*	8.96

TABLE II.—*Analyses of milk*—Continued.

Sp. gr. at 15.5°.	Total solids.	Fat.	Solids not fat.	Solids not fat by—			
				Babcock.	Fleischmann.	Hehner and Richmond.	Richmond.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1.0316	12.62	3.77	8.85	9.02	8.92	8.63	*8.80*
1.0315	12.36	3.51	8.85	8.93	*8.84*	8.57	8.72
1.0321	12.06	3.19	8.85	9.03	8.92	8.68	*8.80*
1.0320	12.64	3.80	8.84	9.13	9.02	8.75	*8.90*
1.0316	12.51	3.67	8.84	9.06	*8.90*	8.63	*8.78*
1.0325	12.30	3.46	8.84	9.18	9.08	*8.81*	8.95
1.0318	12.59	3.76	8.83	9.07	8.95	8.69	*8.83*
1.0320	12.48	3.66	8.82	9.10	8.98	8.72	*8.86*
1.0320	12.16	3.35	8.81	9.03	8.93	8.68	*8.81*
1.0323	12.27	3.48	8.79	9.14	8.94	*8.78*	8.91
1.0325	12.30	3.52	8.78	9.22	9.07	*8.81*	8.95
1.0314	12.30	3.52	8.78	8.90	*8.82*	8.55	8.70
1.0324	11.91	3.14	8.77	9.09	8.98	*8.76*	8.85
1.0313	12.53	3.77	8.76	8.94	8.84	8.56	*8.72*
1.0324	12.25	3.49	8.76	9.16	9.06	*8.80*	8.93
1.0326	11.74	3.00	8.74	9.12	9.01	*8.77*	8.88
1.0324	11.73	2.99	8.74	9.06	8.94	*8.72*	8.82
1.0313	12.47	3.76	8.71	8.94	8.84	8.56	*8.72*
1.0317	12.25	3.56	8.69	9.00	8.91	*8.63*	8.79
1.0308	12.75	4.10	8.65	8.87	8.79	8.50	*8.67*
1.0310	11.95	3.31	8.64	8.76	*8.68*	8.41	8.56
1.0299	12.64	4.13	8.51	8.65	*8.57*	8.36	*8.45*
1.0309	11.34	2.88	8.46	8.64	8.56	8.32	*8.44*
1.0255	19.34	10.91	8.43	8.92	8.57	8.27	*8.47*
1.0294	12.06	3.78	8.28	8.44	8.34	8.04	*8.28*
1.0287	9.72	2.05	7.67	7.79	7.84	*7.63*	7.73
1.0234	9.82	3.21	6.71	*6.75*	6.84	6.46	6.75
1.0260	10.17	2.92	7.25	7.37	7.34	7.08	*7.24*
1.0258	10.06	2.93	7.13	7.33	7.29	7.04	*7.15*
1.0141	5.57	1.56	4.01	3.98	*4.02*	3.81	3.96

IS THERE ANY LOSS OF FAT BY THE SOURING OF MILK?

One of the difficulties of analyzing sour milk, especially the estimation of fat therein, is its mechanical condition. After milk has soured and coagulated it is usually hard to get sour milk so thoroughly and evenly mixed that the small quantity necessary for analysis is a fair portion of the whole sample. In order to obviate this difficulty, and at the same time estimate the fat in sour milk, the writer measured several portions of one sample of sweet milk into Babcock test bottles, 17.6 cc. of the milk into each test bottle. This was done with two different samples of sweet milk. A determination of the fat in each sample was made by the Babcock process while the milk was sweet. The other test bottles which contained the same milk were left in a warm room. The milk in these bottles soured and became very rotten before some of them were tested. The sweet milk was measured into all the test bottles April 4. Two tests for fat were made of each sample on April 17, April 26, May 29, and July 26, with the following results:

Date.	Condition of milk.	Per cent of fat.	
		No. 1.	No. 2.
Apr. 4	Sweet	4.8	5.5
17	Sour	4.9	5.5
26	Very sour	4.85	5.5
May 29	Yellow rotten	4.9	5.6
July 16	do	4.8	5.2

With the exception of sample No. 2, tested July 16, the fat which separated in the neck of the test bottles was clear and of the same yellow color in every test, although the milk in the bottles was from two weeks to three months old when tested. The fat which separated in sample No. 2, on July 16, was very dark colored, and the result obtained was lower than that found in the sweet milk. These figures show that in these samples practically the same per cent of fat was found by the Babcock method in sour rotten milk three months old as the milk contained when it was sweet.

Another sample of sweet milk was then analyzed gravimetrically. About 5 grams of milk was weighed into clean dry sea sand in a small porcelain dish and also into asbestos in a perforated metal cylinder. These were dried in a water oven, solids weighed, and then extracted with ether to determine the fat. Several portions of the same sweet milk were also weighed into sand and asbestos, as already described. These were left in a warm room, protected from dust, and the milk allowed to sour in the weighed dishes.

The per cent of solids and ether extract was determined in one of these sour samples about every two days with the following results:

Date	Condition of milk.	Per cent solids.		Per cent ether extract.	
		Milk dried on—		Milk dried on—	
		Sand.	Asbestos.	Sand.	Asbestos.
Mar. 17	Sweet	13.38	13.21	3.83	3.86
19	Sour	12.13	13.20	3.70	3.64
20	do	10.69	13.23	3.58	3.85
21	do	10.18	13.06	3.32	3.27
22	do	10.15	13.17	2.38	3.39
30	do	9.91	12.95	2.25	3.55

The results show that there was a constant decrease from day to day in the percent of solids and ether extract found in the milk weighed and allowed to sour on the sand, while in the same milk which was weighed on asbestos and treated in the same way as that on sand there was a much smaller decrease.

This difference in results may be explained by the way the milk spread out on the sand and asbestos. In the latter case the milk probably dried much more and quicker while standing in the warm room before analysis than it did on the sand in an evaporating dish, so that the fermentation of the milk was less, because there was less water present. If the ether extract obtained in these analyses represents all the fat there was in these samples, the results indicate that there may be a loss of fat by the souring of milk, but on the other hand the same per cent of fat was found by the Babcock process in milk so sour that it was rotten, as the milk contained when it was sweet.

Briefly summarized the work of your reporter on dairy products this year has been:

1. The abstracting of the reports made to this convention in the past by the various reporters on dairy products.

2. The suggestion of a few points for investigation by the members of this Association and sending plain instructions for obtaining the evidence in a printed circular letter to 50 American chemists. In response to this request your reporter had received, up to August 11, one analysis of 1 sample of milk from 1 American chemist.

3. Observations on the specific gravity of 6 samples of milk at intervals of one to twenty-four hours after the milk was drawn from the cows. These showed nearly the same specific gravity when the milk was fresh as when it was twelve to twenty-four hours old.

4. Duplicate gravimetric analyses of 15 samples of milk, including the estimation of solids and fat by the sand and asbestos methods, which gave practically identical results. A comparison of the per cent of solids so obtained with that found by using Fleischmann's, Babcock's, Hehner & Richmond's, and Richmond's formulas for calculating the solids in milk from its specific gravity and per cent of fat. In every case the per cent of solids so obtained was nearest to that found by weight, when this calculation was made by using the formulas of Hehner & Richmond.

5. Practically the same per cent of fat was found by the Babcock method in very sour milk three months old as the milk contained when sweet, but less solids and ether extract was found in milk which was mixed with sand and allowed to sour before it was analyzed than in the same milk when it was sweet.

In conclusion your reporter wishes to express his thanks to Mr. H. Droop Richmond, of England, for the valuable contribution he has made to this report, and his sympathy for the many American agricultural chemists who have indicated by their lack of returns that they are either thoroughly posted on the points which they were requested to investigate, or that the arduous duties of their official positions compelled them to only "look and long" for the necessary time to do such work.

Since writing this report the following papers have been received:

THE DETERMINATION OF ALBUMEN IN COW'S MILK.

By L. L. Van Slyke.

At our last meeting I presented a paper * on the determination of casein in cows' milk. I desire now to present a paper supplementary to the one read last year.

Ordinarily, when we speak of milk albumen, we mean the portion of nitrogen compounds not coagulated by rennet, acids, and certain salts. In other words, we apply the term albumen to the nitrogen compounds left after removing the casein proper. This use of the term albumen is inaccurate, because, after removing from normal milk its casein, there remain at least two classes of nitrogen compounds. One of these is coagulated by heat, especially in presence of dilute acids, while the other compound or class is not coagulated under these conditions. To the former of these two compounds or classes the term albumen is properly applied.

DETAILS OF METHOD.

The filtrate obtained after separating the casein by the method described last year is placed in a water bath heated to the boiling temperature of water, the beaker containing the filtrate being covered with a watch-glass crystal. The albumen is completely precipitated under these conditions in ten or fifteen minutes, when the coagulated albumen settles to the bottom of the beaker, leaving the supernatant liquid clear.

The precipitate is filtered, washed, and then treated according to the Kjeldahl method for determining nitrogen. The amount of nitrogen multiplied by the factor 6.25 gives the amount of albumen.

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*Bulletin No. 38, U. S. Department of Agriculture, Div. of Chem., p. 109.

In order to ascertain whether the length of time of heating the solution containing the albumen influenced the results, the digestion was varied from five minutes to ten hours. The tabulated results are as follows:

Sample of milk.	Length of time solution was heated.									
	5 minutes.	10 minutes.	15 minutes.	20 minutes.	30 minutes.	45 minutes.	1 hour.	2 hours.	4 hours.	10 hours.
	Per cent of nitrogen in albumen contained in milk.									
No. 1:										
a	....	0.060	....	0.057	0.054	0.056	0.057	0.061	0.046	0.060
b	....	.062	0.058	.048	.051	.063	.062	.059	.055	.054
Average	....	.061	.058	.053	.053	.059	.059	.060	.051	.057
No. 2:										
a	0.014	.054	.054	.052	.052	.051	.052	.047	.045	......
b	.044	.053	....	.054	.051	....	.051	.051	.049	......
Average	.029	.054	.054	.053	.052	.051	.052	.049	.047	......
No. 3:										
a	.054	.053	.049	.052	.051	.053	.057	.057	.056	.058
b	.054	.047	.053	.056	....	.057	.057	.060	.053	.055
Average	.054	.050	.051	.054	.051	.055	.057	.059	.055	.057
No. 4:										
a	.057	.064	.061	.061	.065	.065	.062	.062	.069	.065
b	.062	.055	.065	.063	.064	.062	.065	.066	....	.065
Average	.059	.060	.063	.062	.065	.064	.064	.064	.069	.065
Average of all results	.047	.056	.056	.055	.055	.057	.057	.058	.056	.059

An examination of the foregoing table shows—

(1) In one case, heating for five minutes gave low results; in two other cases, good results.

(2) In general, the results varied very little with increased length of heating.

(3) There was a slight tendency to higher results with increased length of heating, but such increase was irregular and, at most, amounted to only 0.002 or 0.003 per cent nitrogen.

(4) It would therefore appear that entirely satisfactory results can be secured by heating the solution under given conditions for ten or fifteen minutes.

The precipitate formed always filters readily and washes easily.

SEPARATION AND DETERMINATION OF THE NITROGEN COMPOUNDS OF COWS' MILK.

1. Total nitrogen compounds.—Determine the amount of total nitrogen compounds by Official method.

2. Casein.—Weigh out about 10 grams of milk, dilute in a beaker with about 90 cc. of water at 40° to 42° C., and add at once 15 cc. of a solution containing 10 per cent of acetic acid, by weight. Stir with a glass rod and let stand three to five minutes longer. Then decant on filter, wash two or three times with cold water by decantation, and then transfer precipitate completely to filter. Wash once or twice on filter. The washed precipitate and filter paper are then digested as in the regular Kjeldahl method for the determination of nitrogen, and the determination completed in the usual manner. To calculate the nitrogen into an equivalent amount of casein, multiply the amount of nitrogen found by the factor 6.25.

3. Albumen.—The filtrate obtained above in separating casein is placed in a water bath and heated to the boiling temperature of water for ten or fifteen minutes. The washed precipitate is then treated by the Kjeldahl method for determining nitrogen. The amount of nitrogen multiplied by 6.25 gives the amount of albumen.

4. Remaining nitrogen compounds.—The remaining compound or compounds of nitrogen are determined by difference, subtracting from the amount of total nitrogen compounds the sum of the casein and albumen.

In conclusion, I wish to call attention to the crude nomenclature in common use in stating the results of milk analysis for nitrogen compounds. It is an almost universal custom to call the total nitrogen compounds of milk casein. It would be quite as correct to call the fat of milk palmitin or some similar name. This wrong use of the term casein is bound to lead to more or less confusion, and I would suggest that our Association adopt and recommend some better form for stating the results of milk analysis with reference to the nitrogen compounds. I believe, also, that the time has come when the Association should take up the study of the methods of separating and determining these different nitrogen compounds of milk. I hoped that the reporter on dairy products might see the way clear to commence such work this year. If there is in the mind of any a tendency to think that the Association has reached such a stage in its study of dairy products as to call for little or no further effort, it is to be hoped that they may be brought to realize the fact that our hardest work lies in the future.

I am much indebted to Mr. A. L. Knisely for assistance rendered by him in carrying out the analytical details of the work.

DEAR SIR: In accordance with your circular note of March 7, I have caused analyses of four samples of milk to be made in the laboratory. No samples of known origin being at my command, the milks examined were bought in the open market. The work was done by Mr. K. P. McElroy. The results obtained appear in the following table:

Analyses of milk.

	No. 13207.	No. 13208.	No. 13209.	No. 13210.
Specific gravity at 15°	1.0278	1.0328	1.0298	1.0291
Solids	10.82	11.24	12.29	13.45
Fat	1.74	2.18	3.44	4.77
Ash	.82	.77	.77	.76
Sugar	3.00	2.92	2.94	3.03
Nitrogen	.57	.58	.57	.54
Albuminoids	3.56	3.63	3.57	3.38

The specific gravity was determined by means of a pyknometer. Ash was determined by the official method, igniting after addition of nitric acid and drying. Sugar was determined by the polariscope. Nitrogen was estimated by the Kjeldahl process.

Water was determined in two ways—by drying in flat platinum dishes containing 1.5 grams of ignited asbestos and drying in Babcock tubes of 2 cm. diameter and 6 cm. height, carrying 2 grams asbestos. The amount of milk used was about 1 gram in each case. Drying was done at 100° in a water-jacketed bath, the dishes and tubes being in immediate contact with its walls. One hour's drying was sufficient to expel the bulk of the water, as may be seen by the appended table:

	Per cent solids.							
	No. 13207.		No. 13208.		No. 13209.		No. 13210.	
	1 hour.	2 hours.	1 hour.	2 hours.	1 hour.	2 hours.	1 hour.	2 hours.
Platinum dish	10.82	10.82	11.14	11.17	12.73	12.47	13.59	13.43
	10.89	10.81	11.26	11.28	12.58	12.58	13.47	13.47
Babcock tube	....	....	11.37	11.40	12.48	12.33	13.47	13.42
	....	....	11.19	11.19	11.97	11.80	13.63	13.49

Fat was determined by extracting the residues from the water determination in a mercury seal extractor for ten hours and drying and weighing the percolate. Anhydrous, alcohol-free ether was used. The Babcock tubes were slipped into glass tubes carrying a plug of asbestos to act as a filter to prevent fine asbestos dust from the tubes being washed down into the fat by the ether. The asbestos in the dishes was transferred to an ordinary extraction tube and the dish washed with ether. The results obtained are as follows:

	Per cent fat.			
	No. 13207.	No. 13208.	No. 13209.	No. 13210.
Dish	1.71	2.01	3.52	4.68
	1.76	1.85	3.41	4.80
Babcock tube	....	2.32	3.44	4.70
	....	2.33	3.40	4.90

Respectfully,

G. L. SPENCER,
Acting Chief of the Division of Chemistry.

Prof. E. H. FARRINGTON,
Reporter on Dairy Products, Association of Official Agricultural Chemists, Champaign, Ill.

DETERMINATION OF THE RISE IN SPECIFIC GRAVITY ON ALLOWING MILK TO STAND AFTER MILKING.

By H. DROOP RICHMOND.

The specific gravity was taken at the expiration of one, six, eighteen, and twenty-four hours after milking.

The determinations at one, six, and eighteen hours were made by means of a lactometer, the temperature noted at the same time; that at twenty-four hours was determined by a Sprengel tube of 10 cc. capacity, and the apparent coefficient of expansion in glass estimated at the same time; the latter was used to correct the lactometer determinations to 15.5° C., and in no case did the result thus corrected differ from the result corrected by means of the table generally used by more than 0.0001. The table which was used in the determinations of specific gravity already reported is thus incidentally shown to be correct.

Total solids and fat determined as before.

Table of results.

	Specific gravity at 15.5° C.				Total solids.	Fat.	Solids not fat.
	1 hour.	6 hours.	18 hours.	24 hours.			
					Per cent.	*Per cent.*	*Per cent.*
	1.0304	1.0304	1.0304	1.0304	13.98	5.04	8.94
	1.0300	1.0299	1.0300	1.0300	13.86	5.00	8.86
	1.0327	1.0328	1.0328	1.0328	14.50	4.79	9.71
	1.0308	1.0311	1.0317	1.0318	14.04	4.90	9.14
	1.0319	1.0318	1.0321	1.0321	13.63	4.37	9.26
	1.0395	1.0396	1.0396	1.0397	11.55	1.04	10.51
	1.0310	1.0322	1.0325	1.0325	14.62	5.30	9.32
A	1.0312	1.0318	1.0319	....	....	4.55	....
B	1.0318	1.0317	1.0319	....	....	4.60	....

The samples marked A and B were from the same cow, taken simultaneously; A was cooled to about 12° C. at once, while B was not cooled. The fat in these is by the Leffmann-Beam method.

In another sample one portion was allowed to stand without any addition, while 0.05 per cent of salicylic acid was added to another.

The specific gravities were:

	½ hour.	1½ hours.	4 hours.	7 hours.	24 hours.	Fat.
Nothing added	1.0313	1.0317	1.0318	1.0317	1.0319	2.70
Salicylic acid	1.0313	....	1.0313	1.0315	1.0319	

The milk sugar, by polarization, was estimated after one-half hour and after nine hours.

The results were:

	Per cent.
½ hour	4.72
9 hours	4.69

This experiment shows that the rise in specific gravity is not due to a change in the milk sugar; other experiments in a different direction fully confirm this and will be shortly published.

Salicylic acid seems to check the rise; it may possibly be due to an enzyme, and perhaps a change in the casein analogous to that of fibrin may take place. Recknagel attributes the property to a swelling of the casein.

The solids not fat calculated from my formula are inserted in the right-hand column in the table of results. The twenty-four hours specific gravity is used for calculation.

It is seen that in the two cases where there was a distinct rise the agreement is good between the found and calculated results; in the others, where the rise is practically nothing, the calculated solids not fat are too low.

I have found that the concordance between solids not fat found and that calculated from any formula fluctuates with the season to a notable extent. It may happen that the rise in specific gravity takes place under certain climatic and other conditions at one season of the year and not at others.

It would be useful to study periodically for a whole year this question.

I may add that until this year I had always found a distinct rise in the specific gravity, and that my former experiments had always been conducted in the autumn and winter.

Messrs. L. K. Boseley and F. W. Joint have made several of the determinations.

DOES CREAM LOSE FAT ON KEEPING FOR A LONG TIME WITH AND WITHOUT BICHROMATE OF POTASH?

By A. L. Winton.

Cream was used for these experiments rather than milk, because with a higher per cent of fat a loss of this ingredient would be more apparent. On April 23 a quantity of sweet Cooley cream containing 22.25 per cent of fat was thoroughly mixed and divided into two samples. To one of the samples bichromate of potash was added in the proportion of 1 gram of the salt to 1 liter of the milk; to the other sample no preservative was added.

A number of portions of 18 cc. each was removed by a pipette from each sample and placed in test bottles for the determination of fat in cream by the Babcock method. Two of these portions, one poisoned and one not poisoned, were tested at once (April 23); the remainder was placed under a bell glass and kept at room temperatures for further tests.

Up to the date of writing tests have been made on four dates at intervals during a period of three months. It is proposed to reserve the remainder of the portions in the test bottles for further tests, running through an entire year. Thus far the portions containing the bichromate of potash look the same as when first prepared, but the others have been for some time in a rotten condition, furnishing a medium for the growth of fungi and maggots. The results thus far obtained follow:

Date.	Per cent of fat.	
	Poisoned.	Not poisoned.
April 23	22.25	22.25
May 2	22.25	22.50
June 11	22.25	22.25
July 22	22.25	22.00

It will be seen that the results, after keeping the cream three months, are essentially the same as those obtained on the fresh samples. The extreme difference of 0.5 per cent, which is probably due to error in pipetting the cream or measuring the fat, is equivalent to less than 1.05 per cent on milk containing 4 per cent of fat.

CALCULATION OF TOTAL SOLIDS IN MILK FROM THE PER CENT OF FAT AND THE SPECIFIC GRAVITY, ACCORDING TO THE FORMULAS OF BABCOCK, FLEISCHMANN, AND OF HEHNER AND RICHMOND, COMPARED WITH THE GRAVIMETRIC METHOD.

By L. L. Van Slyke.

[Abstract.]

Mr. Van Slyke has determined the total solids in milk by the formulas mentioned above in 483 samples of milk and 29 samples of whey.

In the gravimetric method drying on sand was practiced. The average results for the 483 samples of milk are as follows:

	Per cent.
Total solids by gravimetric method	13.12
Total solids by formula of Hehner and Richmond	13.09
Total solids by formula of Fleischmann	13.39
Total solids by formula of Babcock	13.46

The tests began on the 15th of January and closed on the 13th of August, 1894. Milk of the following breeds of cows was used, viz: Ayrshire, Holstein-Friesian, American Holderness, Shorthorn, Guernsey, Jersey, and Devon.

It is seen that the average by the Hehner and Richmond formula is practically the same as that obtained by the gravimetric method, while the average results obtained by the formulas of Fleischmann and Babcock are about 0.3 and 0.4 per cent too high, respectively.

The variations in individual cases, however, are much greater than this. In the case of a Guernsey milk, while the gravimetric method gave 19.2 per cent total solids, the Fleischmann formula gave 18.06 per cent and the Hehner and Richmond 17.70 per cent. In most cases, however, the agreement between the gravimetric method and the results calculated by the different formulas was reasonably close.

Fractional milking was practiced in some cases, so as to vary the percentage of fat in the milk, but in these cases also the agreement was fairly satisfactory.

The same is true also in the case of whey where the percentage of fat was very low, but in these cases the total solids as calculated by the formulas were uniformly higher than those obtained by drying, and this difference was about 0.75 per cent.

The formulas may be regarded as being applicable with a reasonable degree of accuracy with the normal content of fat as found in the milk. With a very high content of fat or a very low content the formulas are less reliable. Inasmuch, however, as these formulas have been devised for the purpose of testing ordinary normal milks, they may be regarded as of considerable utility in this respect, so that when the specific gravity has been accurately determined and the percentage of fat obtained the total solids can be computed with reasonable certainty by the application of the formulas in question. The formulas are as follows:

Let t = per cent total solids, S = specific gravity at 15.5° C. (15 C. in Fleischmann's formula), and f = per cent fat.

Fleischmann: $t = 1.2f + 2.665 \frac{100S - 100}{S}$

Hehner and Richmond: $t = \frac{f + .2186S}{.859}$. If $\frac{S}{t} > 2.5$ correct the value obtained for t by adding $.05 \left(\frac{S}{t} - 2.5\right)$

Babcock: Solids not fat $= \left(\frac{100S - Sf}{100 - 1.0753Sf} - 1\right) \times (100 - f)2.6$

A METHOD OF DETERMINING BUTTER FAT IN MILK OR CREAM BY DIFFERENCE.

By S. W. McKeown.

For several years I have employed the following method of milk analysis with good results:

Weigh as quickly as possible, by means of a sensitive analytical balance, about 2 grams of milk on a platinum crucible lid of 1½ to 2 inches diameter; silver or nickel will answer equally well. Evaporate in the usual manner at a temperature of 212° F. until weight of residue is constant. After taking the final weight, immerse the dish and contents in a beaker containing about 150 cc. of petroleum naphtha; place the lid in an upright position and if necessary add enough naphtha to completely immerse the dish. Leave for one hour or more (I sometimes keep the dish in the naphtha ten or twelve hours) then take out and dry for thirty minutes at 212° F., cool in a desiccator and weigh again. The loss in weight is butter fat. The lid may be again immersed in the naphtha and the operation repeated, but there is rarely any further loss.

Two grams appear to be the proper amount, 5 grams requiring too long a time to evaporate to complete dryness and the residue can not be so thoroughly and so surely exhausted of its fat. I use a silver crucible lid which is flat, and during the evaporation if any blisters appear I puncture them with a platinum wire. Of course some care is necessary to avoid loss of nonfatty particles, but if any such should become detached they can easily be noticed floating in the naphtha. The results agree very closely with those obtained by the Adams method. Less skill and less personal attention are required than by any other method that I know of. The plan of exhausting the residue of its fat by repeated use of small portions of ether or naphtha, or carbon bisulphide, I have found to be tedious and uncertain in its results.

On motion, the report on dairy products was adopted.

REPORT ON TANNIN.

By W. H. Krug.

The Congress of Chemists held in conjunction with the Columbian Exposition at Chicago last year brought together a large number of prominent chemists, among whom were several that are connected with the leather trade. I was at that time engaged at the exhibit of the Chemical Division in the Government building in an investigation of the foreign tanning materials which were exhibited at the Exposition. This work was undertaken for the Forestry Division of the Department of Agriculture, and it was desired to show the practical value of these materials. For this purpose I used the method recommended by Proctor in England which is known to all of you and consists in percolating the liquid through a column of hide powder and determining the solids remaining in the percolate after rejecting the first 50 cc. I was assisted in this work by Prof. Collingwood, of the Arizona Experiment Station, and neither of us was satisfied with the method.

In conjunction with Mr. Hurty and Mr. Westenfelder we extensively discussed the subject, and fully realizing the importance of having a uniform method which, even if not giving absolute results, would at least yield such as were concordant and comparable, we decided to attempt a realization of this. In accordance with this decision a meeting was called for the 30th of October at the rooms of the Chicago Academy of Sciences, and invitations were sent to a number of tanning chemists. Owing to the distance that separates Chicago from the East, where many of these chemists are located, none of the Eastern chemists could be present, but all sent answers expressing manifest pleasure in the fact that such work was to be undertaken and promising their hearty cooperation. Messrs. Hurty, Fiebing, Westenfelder, and myself were present, and after long discussion decided to organize the Association of American Leather Chemists and drafted a provisional method, as follows:

PROVISIONAL METHOD.

1. Use such an amount of extract that 100 cc. of the solution shall contain about 1 gram of dry solids.

2. Add to the extract taken about 200 cc. of water at or over 80° C., stir until dissolved, transfer to a graduated flask, washing out the beaker with warm water, cool and make up to the mark. Shake thoroughly, immediately take 100 cc., evaporate, dry to constant weight at 95 to 100° C., and weigh. Result is total solids. Now filter the remainder of the solution through thick filter paper 9 cm. in diameter (S. and S. sirup filter paper is recommended), using a teaspoonful of purified kaolin if necessary. Take 100 cc. of filtrate, evaporate and dry for total solid matter.

NOTE.—The dishes should be covered during drying, so as to prevent loss by popping, and the residues must always be dried to constant weight, at a temperature not exceeding 100° C.

3. Determine the amount of soluble matter in the hide powder by making a blank analysis identical with the method used for determining the tannin.

4. Take 6 grams of hide powder, put into a glass with 100 cc. of water, and shake for five minutes. This will remove traces of soluble matter. Strain through well-washed muslin or linen, and squeeze out very thoroughly.

Divide the moist hide powder into three portions. Take about 130 cc. of filtered tannin liquid and shake up with the first portion of hide powder for about five minutes. Strain through well-washed muslin or linen. To the strained liquid add the second portion of hide powder, shake again for five minutes and strain as before. To the filtrate add the last portion of hide powder and shake again for about eight minutes. Now filter through sirup filter paper, pouring the liquid back if necessary, so as to obtain a perfectly clear filtrate. Take 100 cc. of the filtrate, evaporate, and dry as before.

(*a*) It is absolutely necessary to use some form of mechanical shaker, as shaking by hand is not intense enough to remove all the tannin. The association recommends any form of the machine known as "milkshaker."

5. Determine the moisture in the extract by drying 2 grams to constant weight.

The method of investigation used by the Association of Official Agricultural Chemists was adopted. This consists in sending to each chemist who is to participate in the work a sample of the substance to be analyzed, together with a minute description of the method to be used. This method is to be closely followed, and the results sent in to the reporter, who tabulates them and draws his comparisons. Uniform samples of extract and hide powder were accordingly sent to the following chemists: Messrs. Hurty, Adriance, Fiebing, Trimble, Westenfelder, Yocum, Collingwood, Kerr, Norris, Trubek, and the Tiffany Chemical Company.

Reports were received from nine chemists.

Table of results.

Analyst.	Total solids.	Soluble solids.	Tanning subst.	Total solids by direct method.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
J. N. Hurty	42.74	41.91	24.88	....
B. D. Westenfelder	41.72	41.22	26.21	42.05
J. H. Fiebing	41.60	40.60	23.20	....
H. Trimble	44.32	41.84	[1]24.90	43.32
W. M. Norris	43.55	40.67	25.75	....
J. S. Adriance	42.98	40.48	23.69	....
J. H. Yocum	43.75	41.66	27.50	....
G. A. Kerr	42.85	41.66	25.20	....
W. H. Krug	41.63	40.46	23.36	42.29
Mean	42.693	41.164	24.974	42.553
Difference between maximum and minimum	2.72	1.45	4.30	1.27

[1] Mr. Trimble's result excluded from the average, not having been obtained by the provisional method.

Residues from 6 grams of hide powder treated by provisional method.

Analyst.	Residues in grams.
J. H. Fiebing	0.008
W. M. Norris	.0149
J. S. Adriance	[2].0043
J. H. Yocum	.040

Analyze [illegible]

[2] This was calculated from result on 5 grams, which was stated to be 0.0715 per cent.

The individual reports differed in so many details that they could not be compiled into one table, and are therefore given below. Mr. Westenfelder, Cincinnati, sends the following report:

TABLE I.

Total solids.	Water by difference.	Total extract.	Soluble extract.	Insoluble extract.	Non-tannins.	Tannins.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
42.05	57.95	41.72	41.22	0.50	14.96	26.26
	....	....	....	....	15.06	26.16
Mean: 42.05	57.95	41.72	41.22	.50	15.01	26.21

TABLE II.—*Determination of nontannins without washing the hide powder.*

Nontannins.	Tannins.	Soluble extract by addition.
Per cent.	*Per cent.*	*Per cent.*
16.87	24.35	41.22
19.13	22.09	41.22
Mean: 18.	23.22	....

Tannin by Löwenthal method, using hide powder to remove the tannin=25.82 per cent.

He writes: "The sample of extract was in a high state of fermentation, which I think will not materially influence the result of tannin, but will lower the figures for solids and nontannins and increase the proportion of tannin to total extractive matter. Please observe that I have given two results for nontannins and tannins. Results in Table I agree, and in Table II disagree. I account for this that I shook No. 2 more violently, thereby causing more soluble matter. At any rate, I can see that if the hide powder is not first washed with water the results will vary as much as by the old filter tube method. As to the results by the Löwenthal method, I will say that I proceeded rather differently from the recommendation given by the German Association of Tanning Chemists. Instead of macerating the hide powder in the liquid for twenty-four hours, I shook up the hide powder in a flask with water, put a plug of absorbent cotton in the neck of a funnel, poured the magma on it, and thoroughly washed it with water. After draining well I poured the liquid upon it and discarded about 50 cc. of the filtrate. The filtrate following was used for titration. I used about 3 grams of hide powder."

Mr. Fiebing, Milwaukee, sends this report:

(1) *Mr. Fiebing's results.*

Extract per liter.	Total solids.	Total soluble solids.	Insoluble matter.	*a.* Nontannins without correction for water in the hide-powder.	*b.* Nontannins with the correction for water in the hide powder.	*c.* Nontannins by filter method.	*d.* Nontannins by shaking with dry hide powder.	*e.* Nontannins by filter method, using different hide powder.	*f.* Nontannins by shaking 24 minutes with total hide powder.	6 grams hide powder contain soluble matter.	6 grams hide powder retain after pressing gram water.
Grams.	*Per ct.*	*Per ct.*	*Per ct.*	*Per cent.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per cent.*	*Grams.*
24.759	41.6	40.7	0.90	17.2	19.2	17.5	19.20	....	....	0.008	14.
24.965	41.6	40.5	1.10	17.6	19.3	17.5	....	17.9	19.5	....	12.
Mean.	41.6	40.6	1.00	17.4	19.25	17.5	19.20	17.9	19.5	.008	13.

(2) *Tannins calculated from the above results.*

a.	*b.*	*c.*	*d.*	*e.*	*f.*
Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
23.20	21.35	23.10	21.40	22.70	21.10

Mr. Trimble, Philadelphia, sends the following report:

Total solids.	Moisture direct.	Insoluble matter.	Soluble extract.	Tannins.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
44.43	56.68	2.52	41.91	24.88
44.21	....	2.44	41.77	24.91
Means: 44.32	56.68	2.48	41.84	24.90

Tannins by the Procter percolation method, rejecting first 80 cc.

	Tannins.
	Per cent.
	21.68
	21.63
Mean:	21.66

He writes: "In every case your suggestions were complied with, except in using a 'milk-shake' apparatus. The hide powder used in the latter method was obtained from Eitner in Vienna."

William M. Norris, Princeton, N. J., writes: "The sample of extract, which stood in a warm place for some time before the analysis was made, fermented."

John S. Adriance, New York, writes: "S. and S. paper No. 591 was used in filtering."

John H. Yocum, New York, sends the following report:

Moisture direct.	Total extract.	Sum of extract and moisture.	Insoluble matter.	Non-tannins.	Tannins.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
56.10	43.75	99.85	2.09	14.16	27.50

Method in use in laboratory.

Nontannins.	Tannins.
Per cent.	*Per cent.*
14.52	27.14

Soluble matter in hide powder, 6 g. to 200 cc.

Official H. P.	Laboratory H. P.
Grams.	*Grams.*
.040	.081

He writes: "The extract was of necessity kept so long that when opened it was found to have soured. While not thinking this would interfere with the analysis, you should know of it. The method in use in the laboratory is the same as the Association method, the only difference being in the hide powder used, and in a slightly longer time in the extraction. The hide furnished by you gave 0.040 grams to 6 grams in 200 cc. of water treated the same way as in an analysis, while that in use in our laboratory gave .081 grams in like amount and method. I would suggest an estimation of the nitrogen as a method of determining the soluble matter in the residue due to the hide powder.

Mr. Kerr, Columbus, Ind., sends the following report:

Method.	Total extract.	Soluble extract.	Insoluble extract.	Tannins.	Water direct.
Provisional method:	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
H. P. from Fiebing..................	42.85	41.66	1.19	25.20	56.57
H. P. from Eitner	42.85	41.66	1.19	25.40	56.57
Laboratory method:					
H. P. from Fiebing..................	42.85	41.66	1.19	24.80	56.57
H. P. from Eitner	42.85	41.66	1.19	25.10	56.57

From Mr. Kerr's letter I infer that he uses the filtering method in his laboratory.

DISCUSSION OF INDIVIDUAL REPORTS.

Mr. Westenfelder sent in three results for tannins obtained by different methods—one by the provisional method, one obtained with unwashed hide powder, and one obtained by the Löwenthal method. The results with unwashed hide powder are much lower than those obtained by the provisional method, but as the latter are not corrected for the water retained by the hide powder it is uncertain whether this difference is due to the omission of the washing, especially as Mr. Fiebing found a difference of but 0.05 per cent between the tannins corrected for the retained water and those obtained with unwashed hide powder. The result obtained by Mr. Westenfelder by the Löwenthal method is 0.4 per cent lower than the provisional method, not an excessive difference.

Mr. Fiebing sent in a very full report. He obtained almost identical results by the provisional method and the filter method. His work on the water retained by the hide powder shows the importance of a correction for this error, the difference between the uncorrected and corrected tannins being 1.85 per cent. He does not state what hide powder he used in obtaining result E, but it is evident by the difference between this and the figure obtained by the filter method, using the official hide powder (Result C.), that the absorptive power of the hides was different, and this is one point which will have to be taken into consideration by the association if it desires to arrive at a satisfactory method. Unfortunately Mr. Fiebing did not make an estimation by the provisional method, using this other hide powder.

Prof. Trimble's report shows the inferiority of the percolation method when compared with the provisional method. It gave a result 3.6 per cent lower than the latter. Prof. Trimble's work, though not done with a milk shaker, compares very favorably with the work of the other analysts, his mean being almost identical with the mean of the figures obtained by the provisional method.

Mr. Yocum uses a method in his laboratory which is almost identical with the provisional method, the only variation being in a slightly longer time of contact with the hide powder. Strangely enough the figure obtained by this method is slightly lower. This again may be due to the hide powder used, having its origin either in a different retentive or absorptive power.

Mr. Kerr reports analyses made by the provisional and filter methods, using Fiebing's and Eitner's hide powder. In both cases the latter gave a slightly higher figure. The results by the filter method are lower, though this difference is not very great.

DISCUSSION OF RESULTS.

In looking at Table No. 1, a compilation of the comparable results, the wide difference between the minima and maxima is immediately evident. In the case of the most important estimation, the tannins, it is absolutely startling and seems beyond explanations.

Various reasons may be advanced for these variations. In the case of the total extract the manner of taking the sample is liable to lead to erroneous results.

Unless an extract is carefully manufactured it often contains material which, even after shaking violently, settles to the bottom before the analyst can take out a uniform sample with the pipette. The fact that several of the analysts allowed some time to elapse before analyzing the sample, so that it was partially fermented, does not seem to have had a tendency to uniformly lower the figure for total extract. Messrs. Westenfelder, Morris, and Yocum reported the sample fermented. The former obtained 41.72 per cent total extract, the two latter 43.55 and 43.75 per cent respectively. The least difference between results is found in the column "soluble extract." The only cause for any difference which can be advanced outside of the destructive influence of fermentation is imperfect filtration. I have found this part of the work quite unsatisfactory. The diluted extract at first filters very rapidly; then the paper becomes clogged, and it takes hours to obtain the quantity requisite to operate with. I think that great improvement may be made at this point. A few analysts sent in reports on the amount of soluble matter in the hide powder treated by the provisional method. They vary so much, however, that it is impossible to explain the variation. The greatest variation is found in the column "Tannins." Various reasons have suggested themselves in explanation of this. They are as follows:

First. The length of time the extract stood in the laboratory before being analyzed. In several cases the analysts reported the sample fermented; in others the fermentation may have commenced, and I do not doubt for a moment that the changes brought about by such fermentation materially affected results.

To test this I analyzed a sample of the extract in July, three months after my first analysis had been made. The sample had stood all this time in a warm place, and the cork was almost driven out by the gases generated during decomposition. On pouring out the extract it was found to be quite thin, whereas the original sample was thick. I obtained the following results:

	Total extract.	Soluble extract.	Tannins.
	Per cent.	*Per cent.*	*Per cent.*
Original analysis	41.63	40.46	23.36
Second analysis	39.83	39.60	21.68
Difference	1.80	0.86	1.68

It is therefore evident that this fermentation does affect the tannins, and we may here seek an explanation for the wide differences exhibited in the table.

Second. Thoroughness with which the diluted extract was mixed with the hide powder, or, in other words, the speed at which the milk shaker is run. This seems to me an important point. The great objection to all previous methods of determining tannin, based on treatment with hide powder, has always been that the liquor did not come in sufficiently thorough contact with the powder to extract all the tannins. The treatment in the "milk shaker" is a most important step towards "eliminating" this objection, but even here a difference in the violence with which the mixture is shaken up must cause some variation.

Third. The amount of water retained by the hide powder after washing and expressing. This is most certainly a source of error, as shown by the results sent in by Mr. Fiebing. His figures show a difference of almost 2 per cent between the results directly determined and those corrected for the water retained by the hide powder. When the hide powder is expressed as directed in the provisional method it is certainly impossible to obtain a uniform retention of water. The statement "squeeze out thoroughly" is, at the best, indefinite, and there will in most cases be a difference of opinion among analysts as to when this is accomplished. I think that the method used by Mr. Fiebing, namely, to squeeze in a copying press, will do much towards diminishing the possibilities of error from this source. The water retained by the hide powder when squeezed can be determined once for all for each batch of hide powder, and this factor applied as the variation between individual determinations when the full pressure of the press is applied is no doubt too small to materially affect the correction.

Fourth. The method of drying the residues. It has been the universal experience in our laboratory that it was well-nigh impossible to arrive at an accurate estimate of the total solids in solutions of organic substances by the direct method. By the direct method, we understand drying in an oven at 100 C. until constant weight is obtained. I say "well-nigh impossible," and might say impossible, because we have never found a substance of this kind that could be dried to constant weight. The determination of the moisture in such materials has been the subject of discussion and experimentation in the Association of Official Agricultural Chemists for many years. All methods of drying have been exhaustively tried by this association, among which may be named, for solids, drying in hydrogen at 100° and drying in air at 100°; for liquids, drying in air, in open dish, at 100°, drying in vacuo at 110°, film drying, etc. The great difficulty with all these is the impossibility of obtaining a constant weight. The samples will lose up to a certain point, and from there on rapidly gain. In experiments made in our laboratory on samples of molasses, with the method of drying in an open dish at 100°, the samples were still losing weight after having been dried one hundred and sixty-five hours. Appreciating the impossibility to obtain absolute final results which represented the true amount of moisture in samples, the agricultural chemists have always used compromise methods, which at least insured comparable figures. Thus, in feeding stuffs they specify drying in hydrogen at 100° for five hours, the loss to be called the moisture; in sugars, sirups, honeys, etc., to dry until the loss in one hour is not greater than 0.20 per cent. There are two reasons for this inability to obtain a constant weight, namely, decomposition and oxidation. Decomposition may set in and cause a loss which is estimated as moisture, and oxidation may begin at some indeterminable time and render it well-nigh impossible to determine the point where the last loss occurred. Both processes may be going on simultaneously, and the result thereby be thoroughly vitiated. At the meeting in Chicago I brought up this point, but was assured at the time that it was extremely easy to obtain a constant weight. I have found, however, in all my work, both with the official sample of extract and a hemlock extract furnished by Mr. Hurty, that the residues would lose for about five hours and then gain very rapidly.

During the last few months Messrs. Carr and Sanborn, of our division, have made extensive experiments on this subject, with the view of solving the problem. It gives me great pleasure to say that they have accomplished this, and, as I am sure that it will interest the association, I will read a letter which they have written at my request and which gives a résumé of their work, together with some of the results achieved.

U. S. Department of Agriculture, Division of Chemistry,
Washington, D. C., August 21, 1894.

Dear Sir: Replying to your request for a description of the drying method with which we have secured such uniform results on readily decomposable sugar solutions, we beg to transmit the following, adapted to meet what we conceive, from your information, to be the primary difficulties in quantitative desiccation of tanning extracts. It is not asserted, much less claimed, that this method is completely satisfactory for the drying of all organic solids in solution. On the contrary, we content ourselves with the belief that it far surpasses any method within our knowledge. We realize very acutely that better results are likely to be obtained by conversion of the decomposable body to be dried into a salt which resists the effect of prolonged heating more completely. In the interim, while researches are under way in the latter direction, we believe it advisable to adopt a method which partially obviates the effect of heating by conducting the drying in a manner which insures maximum rapidity. Drying upon sand, kieselguhr (tripoli), and similar media which depend for their efficiency upon the greatness of heating surface and the division of the drying solution into delicate films, fail because of their tendency to form clots or thick masses which dry very slowly toward the end of the desiccation.

To obviate these difficulties we prepare our drying medium as follows: Good porous pumice stone is broken into pieces (1) approximately 1 cm. square and (2) smaller particles which pass through a sieve having six meshes to the inch, but resist passage through a sieve one size smaller. The smaller particles must be thoroughly freed of dust.

A platinum drying dish of any convenient size may be employed, provided it contains sufficient stone to completly absorb from 5 to 8 cc. of the extract solution.

The dish having been prepared by covering the bottom with the fine particles, and these in turn by a layer of the large pieces, the dish and contents are heated to redness for fifteen or twenty minutes, cooled and weighed. The solution is weighed from a tared bottle or flask, and by means of a pipette with a fine nozzle is evenly distributed over the larger pieces of stone. The object in using the fine particles of pumice stone is to prevent the solution from coming in contact with the dish. It is likely, judging from results upon refractory molasses samples, that the drying will be complete at the end of three hours. We would suggest resort to careful checking as to time of drying. If the solution is fairly homogenous the above weight will suffice.

As an example of the results obtained by the method upon solutions which can not be dried by ordinary methods, we would instance the following upon a prepared solution which contained sucrose, dextrose, levulose, tannic acid, gum arabic, and sodic chloride, the theoretical solids being 23.63 per cent:

Hours drying.	Per cent solids (*a*)	Per cent solids (*b*).
1	23.87	23.93
2	23.76	23.67
3	23.72	23.66
4	23.70	23.72
5	23.65	23.65
6	23.63	23.64

These results are quoted impartially from a great number obtained on the same solution, and are intended solely to convey an idea of the extreme rapidity of the method, which explains in great part, we believe, the possibility of securing uniform results upon solutions containing high percentages of unstable substances.

Trusting that this method may meet the difficulties now encountered in the examination of tanning extracts, we remain,

Very sincerely yours,

O. CARR,
T. F. SANBORN.

Mr. W. H. KRUG,
Reporter.

The fact that the result depends greatly on the manner of drying has been appreciated by H. R. Procter, one of the most prominent leather chemists in England. In an article published in the Journal of the Society of Chemical Industry, **11**, 329, on the "Technical analysis of tanning materials," he describes his method as follows: "Porcelain dishes, 7.5 cm. in diameter, are recommended, and the residues are to be dried at 110° C. and weighed every ten minutes until constant weight is obtained. For determining the water a separate portion is dried at 110° C. Of block gambier and fluid extracts only 1 gram must be used, and this evenly distributed in a thin layer in the dish."

It is evident that where a chemist is limited in time this method of drying is very tedious. He will be continually occupied weighing, and when, as in the case of the extract of a canaigre root, which I recently analyzed, it takes eighteen hours before the residue ceases to lose, weighings every ten minutes are very impracticable. In all technical work speedy and time-saving methods are most desirable, and if it is found that by the method of Messrs. Carr and Sanborn we can obtain reliable results we will have attained this.

I do not think it advisable for us to launch forth into a new method and immediately totally abandon the old. It will be much better to make comparative determinations during the ensuing year, and then at the next meeting settle upon some definite method if the results warrant it. The direct drying of a weighed amount of the extract has its great drawbacks. The extract is thick, and it is difficult to dry it quickly after the surface has become covered with a hard film. I therefore suggest in my recommendations a method modeled after the Association method for sweet wines.

It does not seem necessary to me to make a total extract determination in the diluted extract and a separate moisture determination in the undiluted sample. It appears to be an unnecessary duplication of work. Which of these can be omitted can be determined by future work.

In accordance with the above, I make the following recommendations:

1. That in paragraph 4 of the provisional method, "squeezed thoroughly" be changed to "squeeze in a copying or other suitable press until no more water is expressed."

2. That "shake up with the hide powder" be changed to "shake with the hide powder in a milk-shake apparatus at the highest possible speed."

3. That a paragraph be introduced to read as follows: "Determine the water retained in the hide powder and correct for the error caused thereby."

4. That the reporter for the ensuing year be instructed to make comparative determinations of the total solids by the direct method of drying the extract at 100° C., by the direct method of drying 100 cc. of the diluted extract at 100° C., by the method of Messrs. Carr and Sanborn of drying 10 grams on pumice stone at 100° C. and determining the time required to obtain constant weight and by the following method: "Five grams of the thoroughly mixed extract are weighed into a suitable dish of 200 cc. capacity, diluted with 100 cc. of warm water, evaporated to dryness on the water bath, and dried in an oven at 100° C. until the residue commences to gain in weight."

5. That the reporter be instructed to experiment with modes of filtration with the view of improving this part of the method.

6. That the Association fix upon some limit for the amount of soluble matter in the hide powder.

7. That the reporter be instructed to experiment with the methods of using the three portions of hide powder without intermediate filtration, and of using all the hide powder at once.

8. That the reporter be instructed to experiment with different hide powders to determine whether they vary in absorption power. The retentive power must each time be determined and corrected for.

In conclusion, I offer a report by Mr. Hurty on some analyses made by several chemists of a hemlock extract which, he states, gave him considerable trouble. The results vary as widely as those on the official sample, and what I have said in the case of the latter applies to them.

Determinations on the same sample of hemlock extract, provisional method, by the gentlemen named. Same hide powder used by all.

	Solid matter.	Soluble solids.	Insoluble matter.	Tannin.	Non-tannin.
Fiebing:	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
First time	53.80	44.40	9.40	27.40	17.00
Second time	53.80	44.50	9.30	26.20	18.30
Krug	51.62	44.78	6.84	25.34	19.44
Westenfelder:					
First time	54.86	....	....	....	18.17
Second time	53.64	45.58	8.06	27.90	17.68
W. B. Taber	55.45	49.90	5.55	31.30	18.60
Hurty:					
First time	54.65	47.32	7.33	28.41	20.00
Second time	55.03	47.97	7.06	27.38	18.90

Fiebing's analyses made July 2, 1894. Krug's analyses made July. Westenfelder's analyses made, first one, July 21, 1894; second one, August 3, 1894. Taber's analyses made May, 1894. Hurty's analyses made, first one, June 25, 1894; second one, June 28, 1894.

The following papers were presented:

NOTES ON THE POINTS OF ERROR IN THE SHAKE METHOD OF DETERMINING TANNIN BY HIDE POWDER.

By J. H. Yocum.

The shake modification of the hide-powder method was originated in my laboratory some two years ago, and has been used constantly since that time, the first published account appearing in the Leather Manufacturer, in April, 1893. Since then it has been adopted as provisional by this association and quite recently been favorably commented upon by Dr. Proctor.

I have made a large number of analyses, 800 or more, by this method, and it has given constant results for tannery work. Its advantages over the filter are less time, less trouble, and availability in a very warm climate, and if not more, certainly fully equal accuracy.

I have endeavored to find the corrections necessary to make the results from this absolute. The corrective points are dilution due to water carried into the analysis by the dampened hide and soluble hide substance getting into the non-tannin filtrate.

The method as used in this laboratory does not essentially differ from the provisional one.

To determine nontannins in a tannin solution, 150 cc. of the liquor is taken and shaken with nine grams of hide in three portions, each shaking lasting ten minutes, filtered through linen the first and second shakings and the last time through paper. The hide is prepared by shaking ten grams of the hide with 200 cc. water for ten minutes and throwing on a filter; it is then squeezed and the surplus water removed. This is best done in a press where the same pressure can be obtained each time. About three grams of the wet hide is dried to determine the moisture, equal to about one gram of dry and the balance used for the analysis as above. By this method of procedure six grams of hide powder are used to extract 100 cc. of the tannin solution, and the shaking having lasted for thirty minutes in all the tannin is completely removed.

To determine the dilution and soluble hide errors, four different hide powders were used, samples of the different kinds that had been used in this laboratory from time to time.

1. Granular, color white, no smell, from E. & A.
2. Fine powder and woolly, color white, slight odor, Dr. F.
3. Fine powder and woolly but dusty, whitish blue, smells some, Dr. F.
4. Excellent woolly conditions, white and smells some, Vienna.

Moisture held by the powders air dry.

1.	2.	3.	4.
Per cent. 12.60	*Per cent.* 11.00	*Per cent.* 13.40	*Per cent.* 15.40

From this we see that even when the filter method is used there is a slight dilution, as the powder is used in the filter air dry.

Moisture held by hide after washing and squeezing.

	1.	2.	3.	4.
To absolute dryness.................. per cent..	64.74	72.46	72.16	65.19
To air-dry condition................. per cent..	59.79	68.53	67.67	58.96
Grams of water held by 6 grams hide absolutely dry when dampened and squeezed...	10.79	12.07	12.02	10.87
Grams of water held by 6 grams air dry.......	.75	.66	.80	.96

The dilution due to water from the squeezed hide can thus be readily corrected, while the slight dilution due to moisture in air-dry hide in the filter method can not be allowed for. However, the greater part of this dilution affects the first 30 cc., which are thrown away and as a point of error is trifling in the nontannin filtrate used for the determination. It has been found that the amount of moisture held by the hide, if pressed the same, is uniform, and the determination of the water held by 3 grs. of wet hide will give that put in the tannin solution.

I have endeavored to obtain some data of the soluble hide error in the following way: Gelatin by boiling with an alkaline solution of permanganate of potash, as in Wanklyn's method of water analysis, gives, according to him, 10 per cent of ammonia. It is, I think, a fair conclusion that the soluble matter in the nontannin filtrate will give proportionately the same amount of ammonia as that which is obtained in the first washing of the hide and which can be weighed. With this in view, I made the following determinations:

By shaking thirty minutes 10 grams of hide with 200 cc. of water and evaporating 100 cc. there was found in the 100 cc. evaporated:

	1.	2.	3.	4.
Solids in grams	0.0550	0.1470	0.1585	0.0587
Ash in grams	.0055	.0110	.01450	.0057
Hide substance grams	.0495	.1360	.1440	.0530
Ammonia produced by 100 cc. in grams by hide.	.0061	.0180	.0220	.0070
Ammonia per cent in hide	12.3	13.2	15.2	13.2
Ammonia in nontannin filtrate 100 cc., in grams	.00072	.00200	.00188	.00088
Ammonia in nontannin filtrate then represents hide in 100 cc grams	.0058	.0151	.0123	.0067
Per cent of the hide in the nontannin filtrate of that soluble on the first shaking of the same hide	11.8	11.1	8.5	12.6

To get these data the following procedure was observed:

Ten grams of hide were shaken with 200 cc. of water filtered through paper, and 100 cc. of the filtrate evaporated and weighed.

Two cc. was diluted with ammonia free water and the albuminoid ammonia determined; the washed hide was then used to determine the nontannins, and 5 cc. of the nontannin filtrate analyzed for albuminoid ammonia.

A duplication of this washing of the hide, etc., the same as above, when, instead of determining the nontannins with the washed hide, it was used as in a determination of the nontannins, using water instead of tan liquor, gave:

	1.	2.	3.	4.
Solids in grams	0.0063	0.0212	0.0170	0.0075
Per cent of this found in nontannin filtrate	92.	71.	78.	90.

Generally speaking the most of the soluble hide to water is also soluble in the nontannin filtrate. I would call special attention to the extent of the removal of this soluble hide by the preliminary washing. It seems to me that in this particular the shake process has a great advantage over the filter and that filter results will be higher in nontannins; in fact hide powders 2 and 3 could not be used in the filter with any degree of accuracy, because of the amount of soluble hide given by them, while it could not be used at all on account of its granular condition, yet the results from all, when corrected and before correction, are consistent.

We will now turn to the actual analysis:

	1.	2.	3.	4.
Grams extract in 500 cc. water	10.520	11.256	10.749	11.379
Grams total solids found	4.455	4.735	4.565	4.830
Grams nontannins found	1.735	1.825	1.720	1.880
Grams nontannins corrected dilution	1.6284	2.0440	1.9264	2.0849
Grams hide correction	.0290	.0755	.0615	.0335
Grams nontannins both corrections	1.8994	1.9685	1.8694	2.0404
Per cent nontannins without corrections	16.49	16.21	16.00	16.47
Per cent nontannins with dilution corrections	18.33	18.17	17.92	18.31
Per cent nontannins with both corrections	18.05	17.40	17.35	17.93
Per cent solids	42.35	42.24	42.46	42.44
Per cent tannins without corrections	25.86	26.03	26.46	25.97
Per cent tannins with dilution corrections	24.02	24.07	24.54	24.13
Per cent tannins with both corrections	24.30	24.84	25.11	24.51

In comparing this method with the filter we have a greater dilution, but one which can be accurately measured. By shaking we remove very largely the soluble hide. In the filter the result depends upon the hide. Having shown that the hide that is soluble in water is also very nearly as soluble in the filtrate, that the tannins do not precipitate very much of this soluble hide during the course of the analysis, it is not to be presumed that the 30 cc. thrown away removes the soluble hide to anything near the extent that washing with 200 cc. of water does. Application of a permanganate and soda solution would give relatively the amount of hide in the filter filtrate, but I have not had the time as yet to apply it. Whatever empirical method is adopted by this Association as a standard will have to have its rules very strictly laid down and adhered to, and I believe that for accuracy, rapidity, and ease of working this method is the best. Further, I have no doubt that with well-defined rules of manipulation and correction, that not only can different operators get the same result with the same hide powder, but also with different hide powders and with hide powders which would make it impossible to do any concordant work with the filter.

"HEMLOCK REDS."

By J. N. HURTY.

Under this title I have an account to give of a few facts and of a few experiments; but the paper will contain very little that is conclusive. It is my hope that something herein will prove of value in some way.

My sample of "reds" was obtained from Mr. W. B. Taber, of Alpena, Mich. He informs me that the substance he sent "must be formed during the evaporation under reduced pressure, for the liquors are clear and free from sediment when they enter the pan. The precipitate appears when the evaporation has slightly advanced, and is at its maximum when the concentration is about one-half done. The reds are separated by subsidence." The sample I received weighed several pounds, and was in lumps about the size of walnuts down to very small pieces. The lumps were damp, but quite firm, and in places upon their surface red patches appeared. Fracture was uneven, the freshly exposed surfaces having a deep blue-black color. When cut with a knife the surfaces showed a metallic luster in places, and in other places a dark brown-red color. The odor was that of wet hemlock bark, and the taste, at first faintly sweet, was followed by a slight astringency and bitterness. The bitter was very transitory. The lumps parted with their moisture easily and quickly, and when dry were without difficulty reduced to a brown-red powder. In distilled water at the ordinary temperature 32.62 per cent of matter was dissolved. The remainder was true reds mixed with a small amount of fiber. Of the matter dissolved 48.68 per cent united with hide, which is equivalent to 15.88 per cent of the original substance. This leather-making matter, being soluble, is most probably occluded tannin. In a 1 per cent solution of hemlock extract 21.68 per cent of matter from the reds was dissolved, a difference in favor of water of 10.94 per cent. In a 2 per cent solution of hemlock extract 14.94 per cent of the reds was soluble, a difference in favor of water of 17.68 per cent. These two experiments make it apparent, as was to be expected, that the reds grow less and less soluable in an extract solution as that solution is made stronger. The tanning substance taken up by hide from the water soluble matter in the reds was 15.88 per cent; in a 1 per cent solution of hemlock extract it was 8.44 per cent, and in a 2 per cent solution of the extract it was 9.26 per cent. This increase of tannin in the 2 per cent solution (although it dissolved less matter from the reds) would indicate that varying strengths of extract solutions will take up from the reds irregular quantities of tanning substances. Further experimentation might, however, show this conclusion to be an error. I have been told of instances in the actual process of tanning where reds which were abundant in the vat at the beginning of the process gradually disappeared, the supposition being that they eventually made leather, or at least entered into the hide and gave weight. My slight study of the problem furnishes no contradiction of this conclusion, but there are good reasons for believing that a copious deposit of reds on the hides retards the tanning process not a little. That reds in the tanning vat might in time—because of change in the liquor —dissolve, I can not deny, but my experiments so far do not corroborate the statement. The reds under examination contained—

	Per cent.
Ash	0.765
Matter soluble in petroleum ether	.208
Matter soluble in acetone	52.290
Matter soluble in alcohol of 0.848 sp. gr. after petroleum ether	67.71

The color of the residues after treatment with alcohol and acetone differ materially. They both were red-brown, but that from acetone was very much the lightest.

No other papers being presented the report was discussed.

Mr. YOCUM did not think that a variation in the speed with which the shaking was carried on would influence the results.

Mr. WESTENFELDER considered this worthy of investigation, and Mr. Yocum agreed to experiment on this point and report at the next meeting.

Mr. YOCUM suggested that all results during the ensuing year be figured to a dry basis.

Mr. KEPICH stated it as his belief that results so figured would not be as valuable and more apt to give an erroneous impression. It was decided to leave this to the option of the members.

Mr. WESTENFELDER said that fermentation would increase the per cent of tannins by destroying the nontannins. He did not think that the tannins would be readily destroyed by fermentation.

The advisability of increasing the quantity of liquor and hide powder used in an analysis and of adding all the hide powder at once was then discussed.

Mr. YOCUM. I think that we should use 10 grams of hide powder and 150 cc. of liquor. The wet hide powder should be weighed after pressing and the water determined in about 3 grams by drying. A correction can then be made for the water retained.

Mr. WESTENFELDER. I think this correction an important one.

Mr. FIEBING'S results show the wide difference caused by this retained water.

Mr. KERR moved that Mr. Yocum's recommendations be adopted. The motion was carried.

Mr. KRUG. I think the subject of the hide powder is a most important one. We should decide upon some limit for the soluble matter, and also determine which form of powder it is best to use.

Mr. YOCUM. I think that we should reject any hide powder which gives more than 0.10 gram of residue when 10 grams are shaken for ten minutes with 200 cc. of water and 100 cc. of the filtrate evaporated to dryness.

Mr. KEPICH. We ought to make the method of testing the hide powder conform to the official method. I would suggest that we shake the hide powder three times with water, using 200 cc. each time, and evaporate 100 cc. of the last filtrate. If the residue amounts to more than 0.01 gram the hide powder must be rejected.

Mr. KRUG. I think Mr. Kepich's method of testing a good one, and move that it is adopted.

The motion was carried.

Mr. WESTENFELDER. I move that the following paragraphs be inserted in the method:

"The hide powder must be kept in a dry place and must be tested once a month.

"The above method is applicable to extracts, barks, and all sweet liquors."

The recommendations of the reporter were then adopted.

The society prepared a letter asking for recognition by the Association of Official Agricultural Chemists, and the appointment of a reporter on tannin by said society.

The next order of business was the report of the abstract committee. It was presented orally by Mr. Frear who, however, will hand it to the Secretary in writing.

On motion, the report was adopted.

Mr. WOLL. I wish to know what the abstract committee is doing and what use its work is to the Association? As matters stand I see little benefit to the Association and doubt the advisability of continuing the committee. I would not like to give the impression that I undervalue the work done by the abstract committee. I consider it a most important one and of the greatest value to the members of this Association, if the plan adopted is carried out. My inquiry was inspired by an apprehension that the short statement of the chairman just given would be all that we should have of the work done by the committee, as was the case last year.

Mr. Frear explained that, owing to the unusual promptness of the secretary in publishing the proceedings last year and the delay of the abstract committee, its report failed of publication. He expected, however, to have it published this year.

Mr. Huston. I certainly hope the abstract committee will not be discharged. I feel that the abstracts made by the reporter have really been of very great convenience, at least to the members of the association in the past, and those of us who are not connected with or situated near large libraries find this a matter of very great value.

Mr. Ross was also in favor of the continuation of the committee. Having made something like 200 abstracts from 15 journals, he was glad also to hear that there was a prospect of some of the work finding its way into the journal, even if late.

The Secretary. The abstracts would doubtless have been published last year if we could have had them in time. The question arose, Should we publish the report promptly or keep it back for the abstracts? I acted in the matter on my own responsibility, and decided to go ahead with the reports, hoping to get the abstracts published as a separate bulletin. In this I was not successful, but I think there will be no doubt about publishing the abstracts as a part of our bulletin. What I desire particularly to say is, that in the Department of Agriculture we have an abstract journal, the Experiment Station Record, which, I believe, it is proposed to make cover a great deal more ground than it does now. Therefore I move that the abstract committee which is to be appointed now be requested to correspond with the authorities of the Office of Experiment Stations, with a view to having our abstracts published monthly in that bulletin, so that they will be more promptly available. In this way the abstracts will be promptly published, and every member of the association can have a copy of the record.

The motion was carried.

The Secretary. I have a communication from the Society of Leather Chemists, asking to be incorporated in the association and that a reporter on tannin determination be appointed. This whole matter is easily within the scope of our constitution, and I am heartily in favor of recognizing this branch of agricultural science and acceding to this request.

Mr. Krug. I am the secretary of this association and would like to say that we are engaged in perfecting a method for determining tannin in various products; that we are not commercial chemists, but are simply trying to perfect analytical methods to be applied to the investigation of important agricultural products.

After some further discussion the request of the leather chemists was granted, and the president instructed to appoint a reporter on that subject.

Mr. Lindsey spoke in favor of having the reporters serve two years instead of one. He considered one year too short a time for a reporter to arrive at definite results.

Mr. FREAR seconded Mr. Lindsey's remarks by suggesting that such a recommendation be made to the president.

The SECRETARY also spoke in favor of the motion. It is undoubtedly true that the burden of the reporter's work is in the first year; he is educating himself while he educates the association, and after that his efficiency would be vastly increased with a minimum of labor to him. He suggested that associate reporters be appointed, whose functions should be merely those of education and in the natural order of promotion they would be the next year's reporters.

Mr. WINTON also spoke in favor of the motion.

The president put the question on the recommendation to the president to appoint reporters for two years.

The motion was carried.

The SECRETARY moved an amendment to the constitution, authorizing the president to appoint associate reporters without defining their duties, but with the understanding that they prepare themselves to take the place of the reporters.

On motion, the matter was referred to a committee consisting of Messrs. Wiley, Ross, and Winton, to report at once. The committee reported unanimously in favor of the amendment, which was adopted.

Mr. FREAR moved the thanks of the association to the Secretary, Assistant Secretary, and Chief Chemist of the Department of Agriculture, also to the authorities of the Smithsonian Institution, for courtesies extended.

The motion was carried.

Mr. LINDSEY moved the thanks of the association to the president for his able and courteous manner of presiding over the meetings.

Put by the vice-president and carried unanimously.

Mr. PAYNE wanted to know if a Canadian official chemist was eligible to membership in the Association.

The secretary thought there was nothing in the constitution to prevent it.

The secretary read informally a communication from the German Kali Works in regard to fixing the valuation of potash salts.

The president-elect (Mr. Huston) asked that the reporters serve another year, Mr. Colby taking the place of Mr. Crampton. He then read the list of associate reporters. The abstract committee was also appointed to serve another year.

The secretary asked for the prompt forwarding by reporters of their amended papers, and warned them that the Department of Agriculture now requires papers to be in shape for final publication when submitted, no changes being permitted in galley proofs, except correction of typographical errors.

Moved by Mr. Van Slyke that the secretary be requested to arrange for headquarters at some hotel.

The secretary suggested to include also in this an application to railroads for rebate or reduced fare.

The motion was carried.

Mr. Ross thought these matters should be referred to the committees on cattle food and feeding, and on fermented and distilled liquors. [Applause.]

The association, at 11:40 a. m., adjourned *sine die* to meet next year at the call of the executive committee.

REPORT OF THE COMMITTEE ON ABSTRACTS.

Mr. President and Members of the Association: Your committee on abstracts beg leave to report that their work for this year followed closely that of last year in scope and plan. Unfortunately, pressure of other duties prevented an early organization of the work; and after the allotment of duties was completed it was found that one member would be unable to serve at all. Finally, when the reports of the individual collaborators came in, it was found that, owing to some misunderstanding, several of our most standard analytical journals were entirely overlooked by the member to whom they had been assigned. For that reason the work is less complete than last year. It is believed, nevertheless, that what has been done will be found helpful to the Association.

The committee desires to express its thanks for most efficient collaboration to Messrs. B. B. Ross, of Alabama; Loughridge and Jaffa, of California, and Fields and Haley, of Pennsylvania.

Owing to the delay in reception of the reports of several of the members of the committee for 1892–'93, and to the unusually early day at which the proceedings for 1893 went to press, it was found impossible to prepare the body of the report for the printer within the time allowed. The printing has, therefore, been deferred, and the abstracts for 1892–'93 are arranged with those of 1893–'94 in systematic order, by subjects.

William Frear,
E. W. Hilgard,
W. C. Stubbs,
W. D. Bigelow,
John P. Street,
Committee.

LIST OF JOURNALS ABSTRACTED

1892-'93.

[Including numbers actually issued between June 30, 1892, and July 1, 1893.]

Journal.	Abstractor.
1. Agricultural Science	*Frear.*
2. Alcool et sucre	*Ross.*
3. American Chemical Journal	*Fields.*
4. American Journal of Science	*Fields.*
5. Analyst	*Woll.*
6. Berichte der Deutschen chemischen Gesellschaft	*Winton.*
7. Biedermann's Centralblatt für Agrikulturchemie	*Frear.*
8. Böhmische Zeitschrift f. Zuckerindustrie	*Ross.*
9. Bulletin de la Société Chimique de Paris (June 30 to July 30, 1892)	*Frear.*
10. Bulletin de l'Association des Chimistes de Sucrerie	*Ross.*

	Journal.	Abstractor.
11.	Bulletin de l'Association Belge des Chimistes	*Ross.*
12.	Chemisches Centralblatt	*Allen.*
13.	Chemical News	*Ross.*
14.	Chemiker Zeitung und Repertorium	*Ross.*
15.	Comptes rendus	*Ross.*
16.	Experiment Station Record	*Allen.*
17.	Jahresbericht für Agriculturchemie	*Winton.*
18.	Journal de l'agriculture pratique	*Frear.*
19.	Journal of the American Chemical Society	*Frear.*
20.	Journal of Analytical and Applied Chemistry	*Winton.*
21.	Journal des fabricants de sucre	*Ross.*
22.	Journal of the Chemical Society of London	*Fields.*
23.	Journal of the Franklin Institute	*Frear.*
24.	Journal für Landwirthschaft	*Woll.*
25.	Journal of the Society of Chemical Industry	*Ross.*
26.	Landwirthschaftliche Jahrbücher	*Winton.*
27.	Landwirthschaftliche Versuchs-Stationen	*Woll.*
28.	Landwirthschaftliches Wochenblatt (Schleswig und Holstein)	*Woll.*
29.	Louisiana Sugar Planter	*Ross.*
30.	Milch-Zeitung	*Winton.*
31.	Revue internationale des falsifications	*Frear.*
32.	Sucrerie indigène et coloniale	*Ross.*
33.	Sugar Cane	*Ross.*
34.	Tidschrift för Landsökonomie	*Woll.*
35.	Tidskrift för Landtmänn	*Woll.*
36.	Vierteljahresschrift über die Fortschritte auf dem Gebiete der Chemie der Nahrungs- und Genussmittel	*Winton.*
37.	Zeitschrift für analytische Chemie	*Ross.*
38.	Zeitschrift für angewandte Chemie	*Winton.*
39.	Zeitschrift für Nahrungsmittel-Untersuchungen	*Woll.*
40.	Zeitschrift des Vereins für Rübenzucker-Industrie	*Ross.*

1893-'94.

1.	Agricultural Science	*Frear.*
2.	Berichte der Deutschen chemische Gesellschaft	*Street.*
3.	Biedermann's Centralblatt für Agrikulturchemie	*Haley.*
4.	Bulletin de l'Association des Chimistes de Sucrerie	*Ross.*
5.	Bulletin de la Société Chimique de Paris	*Bigelow.*
6.	Chemisches Centralblatt	*Street.*
7.	Comptes rendus	*Bigelow.*
8.	Chemiker Zeitung und Repertorium	*Street.*
9.	Journal of the American Chemical Society	*Street.*
10.	Journal of the Chemical Society (London)	*Fields.* *Bigelow.*
11.	Journal of the Franklin Institute	*Street.*
12.	Journal für Landwirthschaft	*Haley.*
13.	Journal of the Society of Chemical Industry	*Street.*
14.	Landwirthschaftliche Versuchs-Stationen	*Haley.*
15.	Landwirthschaftliche-Jahrbücher	*Haley.*
16.	Milch-Zeitung	*Street.*
17.	Revue internationale des falsifications	*Frear.*
18.	Vierteljahreschrift über die Fortschritte auf dem Gebiete der Chemie der Nahrungs- und Genussmittel	*Bigelow.*
19.	Zeitschrift für angewandte Chemie	*Bigelow.*
20.	Zeitschrift für Nahrungsmittel-Untersuchungen	*Bigelow.*

LIST OF ABBREVIATIONS USED.

1.	Agric. Science	Agricultural Science.
2.	Alc. sucr	L'Alcool et de sucre.
3.	Amer. Chem. Jour	The American Chemical Journal.
4.	Amer. Jour. Sci	American Journal of Science.
5.	Ann. Chem	Liebig's Annalen der Chemie.
6.	Ann. chim. phys	Annales de chimie et de physique.
7.	Ann. de l'École polyt. Delft.	Annales de l'École polytechnique Delft.
8.	Apoth.-Ztg	Apotheker-Zeitung.
9.	Arch. Hyg	Archiv für Hygiene.
10.	Arch. Pharm	Archiv der Pharmacie.
11.	Atti R. Acc. d. Lincei.	Atti della Reale Accademia dei Lincei.
12.	Bay. Brauer. Jour	Bayerisches Brauerei Journal.
13.	Ber. chem. Ges	Berichte d. Deutschen chemischen Gesellschaft, Berlin.
14.	Ber. Oesterr. Ges	Berichte d. Oesterreichischen chemischen Gesellschaft.
15.	Ber. Ver. Bay. angew. Chem.	Berichte der freien Vereinigung Bayerische Vertreter der angewandten Chemie.
16.	Böhm. Ztschr. Zuckerind.	Böhmische Zeitschrift für Zuckerindustrie.
17.	Bull. Acad. Roy. Belgique.	Bulletin de l'Académie Royale de Belgique.
18.	Bull.Assoc.Chim.Belge.	Bulletin de l'Association Belge des Chimistes.
19.	Bull. Assoc. Chim. France.	Bulletin de l'Association des Chimistes de Sucrerie et de Distillerie de France.
20.	Bull. Soc. Chim	Bulletin de la Société Chimique de Paris.
21.	Bull. Soc. Roy. Pharm. Brux.	Bulletin de la Société Royale de Pharmacie de Bruxelles.
22.	Centralbl. Agrik.Chem.	Biedermann's Centralblatt für Agrikulturchemie.
23.	Chem. Centralbl	Chemisches Centralblatt.
24.	Chem. News	Chemical News.
25.	Chem. Ztg	Chemiker Zeitung.
26.	Chem. Ztg. Rep	Chemiker Zeitung und Repertorium.
27.	Compt. rend	Comptes-rendus hebdomadaires des séances de l'Académie des Sciences.
28.	Conn. Sta. Bull	Bulletin of the Connecticut Agricultural Experiment Station.
29.	D. Zuckerind	Deutsche Zuckerindustrie.
30.	Dingl. polyt. Jour	Dingler's polytechnische Journal.
31.	Exp. Sta. Record	Experiment Station Record.
32.	Gazz. Chim. Ital	Gazzetta chimica italiana di Palermo.
33.	Helfenberg Annalen	Helfenberger Annalen.
34.	Ind. lait	L'Industrie laitière.
35.	Jahresber. chem. Untersuch. Breslau.	Jahresberichte d. chemischen Untersuchungen d. Stadt Breslau.
36.	Jahresber. Stammer	Jahresbericht über die Fortschritte der Zuckerfabrikation.
37.	Jour. agric. prat	Journal d'agriculture pratique.
38.	Jour. Am. Chem. Soc	Journal of the American Chemical Society.
39.	Jour. Anal. Chem	Journal of Analytical and Applied Chemistry.
40.	Jour. Chem. Soc	Journal of the Chemical Society of London.
41.	Jour. fabr. sucr	Journal des fabricants de sucre et des distillateurs.
42.	Jour. Frank. Inst	Journal of the Franklin Institute.
43.	Jour. f. Landw	Journal für Landwirthschaft.

44. Jour. pharm. chim	Journal de pharmacie et de chimie.
45. Jour. prakt. Chem	Journal für praktische Chemie.
46. Jour. Russ. Chem. Soc.	Journal of the Russian Physical and Chemical Society, St. Petersburg.
47. Jour. Soc. Chem. Ind.	Journal of the Society of Chemical Industry.
48. Jour. Soc. Pharm. d'Anvers.	Journal de la Société de Pharmacie d'Anvers.
49. Jour. Zuckerind	(?)
50. Kgl. Landtbruks-Akademiens Handlingar.	Kongliga Landtbruks-Akademiens Handlingar och Tidskrift. Stockholm.
51. Landw. Vereinsschr. Balt. Cent.-Ver.	Landwirtschaftliche Vereinschrift des Baltischen Central-Vereins.
52. Landw. Vers.-Stat	Landwirthschaftliche Versuchs-Stationen.
53. La. Planter	The Louisiana Planter and Sugar Manufacturer.
54. Maandbl. voor Natuurwetensch.	Maandblad voor Natuurwetenschappen.
55. Milch-Ztg	Milch-Zeitung.
56. Miss. Sta. Bull	Bulletin of the Mississippi Agricultural Experiment Station.
57. Mitth. kgl. tech. Versuchss. Berlin.	Mittheilungen der königlichen technischern Versuchs-Station zu Berlin.
58. Molk. Ztg	Deutsche Molkerei-Zeitung.
59. Monatsh. f. Chem	Monatshefte für Chemie und verwandte Theile anderer Wissenschaften.
60. Monit. Scient	Moniteur Scientifique.
61. Neue Ztschr. Rübenzucker.	Neue Zeitschrift f. Rübenzuckerindustrie.
62. Oesterr.-ungar. Ztschr. Zuckerind.	Oesterr.-ungarische Zeitschrift f. Zuckerindustrie und Landwirthschaft.
63. Pflüger's Archiv	Archiv für die gesammte Physiologie des Menschen und der Thiere.
64. Pharm. Centralbl	Pharmaceutisches Centralblatt.
65. Pharm. Centralh	Pharmaceutische Centralhalle für Deutschland.
66. Pharm. J. Trans	Pharmaceutical Journal and Transactions.
67. Pharm. Rev	(?)
68. Pharm. Ztg	Pharmaceutische Zeitung.
69. Pharm. Rundschau	Rundschau für die Interessen der Pharmacie, Chemie und der verwandten Fächer. Prague.
70. Proc. Amer. Acad	Proceedings of the American Academy of Science.
71. Rev. internat. falsificat.	Revue internationale des falsifications.
72. Rev. Internat. Sci. Fals. Aliment.	(?)
73. Schw. Woch. Chem. Pharm.	Schweizerische Wochenschrift für Pharmacie.
74. Selmi, Francesco	Annali chimica Italiano.
75. Staz. sper. agr. Ital	Le stazioni sperimentali agrarie Italiane.
76. Sucr. Belge	Sucrerie Belge.
77. Sucr. indig	Sucrerie indigène et coloniale.
78. Tr. Inst. Brewing	Transactions Institute of Brewing.
79. Trav. Chim. d. Pays-Bas.	Travaux chimiques des Pays-Bas.

LIST OF ABBREVIATIONS USED—Continued.

80. Vjschr. Chem. Nahr....	Vierteljahresschrift über die Fortschritte auf dem Gebiete der Chemie der Nahrungs- und Genussmittel.
81. Wis. Sta. Bull........	Bulletin of the Wisconsin Agricultural Experiment Station.
82. Wien. Wochenbl........	Wiener Wochenblatt.
83. Ztschr. anal. Chem	Zeitschrift für analytische Chemie (Fresenius).
84. Ztschr. angew. Chem ..	Zeitschrift für angewandte Chemie.
85. Ztschr. anorg. Chem ...	Zeitschrift für anorganische Chemie.
86. Ztschr. Nahr. Untersuch.	Zeitschrift für Nahrungsmittel-Untersuchung und Hygiene.
87. Ztschr. physiol. Chem.	Zeitschrift für physiologische Chemie.
88. Ztschr. allgem. Oesterr. Apotheker-Verein.	Zeitschrift des allgemeinen österreichischen Apotheker-Vereins.
89. Ztschr. ges. Brauwesen.	Zeitschrift für das gesammte Brauwesen.
90. Ztschr. Ver. Rübenzucker-Ind.	Zeitschrift des Verein für Rübenzucker-Industrie.

FERTILIZER ANALYSES—GENERAL.

1. TERNE, BRUNO, October 18, 1892.—*Report of the Proceedings of the Ninth Annual Meeting of Official Agricultural Chemists, held at Washington, August 25–27, 1892, with special reference to the methods adopted for the control of the fertilizer trade.*

After recounting the history of the organization of the association, Dr. Terne presented quite fully the results presented by the reporters on potash, nitrogen, and phosphoric acid. The author regards the Lind-Gladding method for potash as very satisfactory, but recalls the difficulties of potash mixing, and consequently of sampling, as noted in his paper in Bulletin 31, Chemical Division, U. S. Department of Agriculture, pages 150–152. He states that the work in nitrogen is most accurate, but that he can not agree with the unofficial chemists who persist in using dilute HCl for the absorption of ammonia and then simply evaporate and weigh the resultant NH_4Cl—a method still followed. In the case of phosphoric acid the results for 1888–'92 are given. The author notes that the association seems to progress backward, but only apparently, since in earlier years the samples were mainly rock goods, while latterly they are more complex. The discrepancies in moisture results are attributed to carelessness (!). In view of the proposition of Gibson (Proceedings for 1890, p. 170) to change the official citrate solution to one slightly alkaline, the author states that he finds them to produce no different results applied to rock goods. Other tests also indicated that an increase of citric acid by 33 per cent made little difference upon the solubility of such goods—less than 0.3 per cent. Neutral citrate, kept for two years in a dark closet in closely stoppered bottles, was found not to have departed from neutrality. The addition of salicylic acid (0.1 gram to 2 liters) is found to prevent molding, without changing solvent action of the citrate. He does not hesitate to say that if the official methods were carefully executed by a competent analyst, such discrepancies as the reporters have shown would not be possible, even in the case of iron and alumina phosphate. These differences exert serious influence upon the fertilizer trade, as is apparent when the resultant difference in valuation is considered.

Jour. Frank. Inst., **134**, 450–467. W. F.

2. OFFICIAL METHODS OF GERMAN EXPERIMENT STATIONS.—Landw. Vers.-Stat., **42**, 97–178; Exp. Sta. Record, **4**, No. 11.

PHOSPHORIC ACID.

GENERAL GRAVIMETRIC METHODS.

A.—AMMONIO-MAGNESIUM PHOSPHATE PRECIPITATION.

3. v. LORENZ, N.—*A source of error in the estimation of phosphoric acid by magnesia mixture (Eine Fehlerquelle bei der Bestimmung von Phosphorsäure mit Magnesia-Mixtur).*

In the absence of citric acid, the simultaneous precipitation of some magnesia (about 1 per cent) can not be prevented. When 2 per cent of citric acid in the form of ammonium citrate is added, and then the magnesia mixture, drop by drop, not a trace of magnesia can be found in the precipitate.

Ztschr. anal. Chem., **32,** 64–67; Journ. Chem. Soc., **64,** ii, 185; Ztschr. angew. Chem., 1893, 232; Ber. chem. Ges., **26,** Ref., 335; Chem. Centralbl., 1893, i, 319; Exp. Sta. Record, **4,** 677; Rev. internat. falsificat., **1,** 44. J. W. F.

4. FOERSTER, O.—*Estimation of phosphoric acid by the molybdate process.*

The adherence of molybdate to the magnesium precipitate will be prevented if the solution is gently heated before adding the magnesia mixture, cooled, and well stirred before filtering. The author washes with a solution containing 2½ per cent of ammonia and 5 per cent of ammonium nitrate.

Chem. Ztg., **16,** 109; Jour. Chem. Soc., **62,** 1519. J. W. F.

B.—LOSS OF PHOSPHORIC ACID ON IGNITION OF AMMONIO-MAGNESIUM PHOSPHATE.

5. NEUBAUER, H.—*Loss of phosphoric acid by the American Official method.—Estimation of phosphoric acid as magnesium pyrophosphate.*

The author states that phosphoric acid is lost during the ignition of the magnesium precipitate, if more than a certain quantity of ammonium salts present during the precipitation, and that he has detected it on a crucible lid coated with a thin layer of magnesium oxid and used to cover the crucible during ignition.

Ztschr. anorg. Chem., **2,** 45–50; Jour. Chem. Soc., **64,** A, 236; Ber. chem. Ges. **25,** Ref. 916; Ztschr. angew. Chem.. 1892, 737; Exp. Sta. Record, **4,** 584. J. W. F.

6. H. NEUBAUER, 1894.—*On the determination of phosphoric acid as magnesium pyrophosphate.*

The author attacks the reliability of the molybdenum method for the determination of phosphoric acid, chiefly because of a loss through the volatilization of the phosphoric acid during the ignition previous to weighing.

Magnesium-ammonium phosphate can be obtained in three different conditions:

(1) The precipitate is formed in neutral or ammoniacal solutions containing no excess of magnesium salt. The precipitate contains less than the normal amount of magnesia and the phosphoric acid is volatilized on ignition; therefore the weights obtained are too low.

(2) The precipitate is formed in the presence of an excess of magnesium salt, but during its formation ammonia is not present in excess. The precipitate possesses the normal constitution, and the weights obtained are correct.

(3) The precipitate is formed in the presence of an excess of both ammonia and magnesium salts. The precipitate contains an excess of magnesia, and the weights are consequently too high.

It appears impossible to completely convert phosphoric acid into pure magnesium pyrophosphate under analytical conditions where the old method is employed. He therefore recommends the following method of procedure: Obtain the yellow precipitate in the usual way and wash it. Dissolve the phosphomolybdate in 100 cc. of cold 2.5 per cent ammonia and add as many centimeters of the usual magnesia mixture as there are centigrams of phosphoric acid present. Addition should not be faster than 10 cc. per minute, stirring constantly. Stir and allow to stand at least three hours. Wash with 2.5 per cent ammonia till the chlorin reaction disappears, dry the filter, and introduce into a well-cleaned crucible which has been thoroughly ignited. Place the lid at an angle and ignite, raising the heat gradually, though not higher than a medium red heat, until the pyrophosphate becomes completely white. Then use the blast and ignite to constant weight. To the weighed amount of pyrophosphate add the correction given in the table below:

Magnesium pyrophosphate.		Magnesium pyrophosphate.	
Found.	Lost.	Found.	Lost.
Gram.	*Milligrams.*	*Gram.*	*Milligrams.*
0.10	0.6	0.24	4.0
.12	.8	.25	4.2
.14	1.2	.26	4.6
.15	1.4	.27	5.0
.16	1.6	.28	5.5
.17	2.4	.29	6.1
.18	2.6	.30	6.8
.19	3.2	.31	7.6
.20	3.5	.32	8.6
.21	3.6	.33	9.6
.22	3.8	.34	10.6

Jour. Am. Chem. Soc., **16**, 289–297; Chem. Ztg., **17**, Repert., 196; Ztschr. anorg. Chem. **4**, 251–266; Ber. chem. Ges., **26**, Ref., 824, 825; Ztschr. angew. Chem., 1893, 610, 611; Jour. Chem. Soc., **64**, A, 489. J. P. S.

C.—ACTION OF AMMONIUM CITRATE ON REVERTED PHOSPHATES.

7. WILLIAMS, W. J., October 18, 1892.—*Discussion of paper of B. Terne on Proceedings of Ninth Meeting, Association of Official Agricultural Chemists.—(Time element.)*

Emphasizes remarks concerning discrepancy in phosphoric acid results. Does not believe that slight difference in acidity of citrate will account for the results, but does believe that a difference in the time of exposure, such as is possible under the present official directions for treating the citrate, will fully account for it.

Jour. Frank. Inst., **134**, 467–469. W. F.

8. GIBSON, C.—*Differences in the determination of reverted phosphoric acid by the American Official method of analysis.—Effect of alkalinity.*

The author endeavors to account for the difficulty by variation in the character of the ammonium citrate solutions. He prepared three solutions as follows: (*a*) According to the official directions; (*b*) by adding ammonia to (*a*) until it showed a very faint alkalinity with litmus paper; and (*c*) made according to H. Joulie's directions, strongly alkaline. In the first series of experiments calcium phosphate was digested with each of these solutions, following the official directions with solutions (*a*) and (*b*) and Joulie's directions with (*c*), but digesting only half an hour, and at the ordinary temperature. In the second series the digestion was continued for an hour. In the third and fourth, aluminum phosphate was used instead of calcium phosphate. The mean numbers obtained, with each of these solutions, for the percentage of reverted phosphoric acid in the first and second, and of available phosphoric acid in the third and fourth series of experiments, were as follows:

	(*a*)	(*b*)	(*c*)
First series	0.913	0.746	0.746
Second series	1.846	.761	.760
Third series	19.77	31.66	26.91
Fourth series	31.39	34.73	34.73

The aluminum phosphate could be almost completely dissolved in all three solutions if sufficient time be given. The author suggests that the American official method be modified to permit of ammonia being added to the ammonium citrate solution until faintly alkaline to red litmus paper, and that the digestion be continued one hour at 65° C., with Joulie's method as an alternative.

Chem. News, **65,** 209, 210, 221; Journ. Chem. Soc., **62,** 1126; Ztschr. angew. Chem., 1893, 27. J. W. F.

D.—CITRATE METHOD.

9. v. LORENZ, N., June 20, 1892.—*The citrate method for phosphoric acid determination* (*Erfahrungen bei der Anwendung der Citrat-Methode zur Phosphorsäure-Bestimmung*).

The results of experiments at several of the experiment station laboratories show that in general the citrate method gives higher figures than the molybdate method. It is known that the precipitate of magnesium-ammonium phosphate, where Märcker's citrate method is employed in analysis of material containing calcium phosphate, always contains a small amount of calcium phosphate. With addition of adequate quantities of ammonium citrate solution the precipitation is not complete, so that in general the two errors balance each other. The first error may be eliminated by removing the lime previous to precipitating the phosphoric acid.

The following method is recommended for the determination of total phosphoric acid in spodium, phosphorite, precipitated phosphate, and even in bone meal and Thomas slag. The reagents employed are: (1) HCl of sp. gr. 1.105; (2) sulphuric acid 1:1; (3) cold saturated solution of ammonium oxalate; (4) acid ammonium citrate solution which contains 400 grams of citric acid and 84 grams of ammonia per liter. Five grams of substance are heated gently with 40 cc. of HCl, avoiding formation of lumps. Then add 20 cc. of dilute H_2SO_4 and boil until the contents of the flask are in a thick pasty condition. Cool and add 250 cc. ammonium oxalate solution, boil again and add 100 cc. acid ammonium citrate solution. After a minute's further boiling, cool, bring the liquid into a 500 cc. flask and, after mixing, filter through a dry filter. Fifty cubic centimeters of the filtrate are mixed with 100 cc. dilute ammonia (2.5 per cent) and precipitated as usual with 25 cc. magnesia mixture. It is claimed that this process yields highly satisfactory results.

Oesterr.-ungar. Ztschr. Zuckerind., 1892, 664–666; Chem. Ztg. **17,** 956; Chem. Centralbl., 1892, ii, 629. B. B. R.

10. JOLLES, A.—*Determination of phosphoric acid* (*Phosphorsäure-Bestimmung*).

Corroborates v. Lorenz's observations on the difference between results by citrate and molybdate method. Describes and gives experience with E. Albert's method and proposes a modification of it.

Oesterr.-ungar. Ztschr. Zuckerind., 1892, 683, 684; Chem. Centralbl., 1892, ii, 630. E. W. A.

11. MÄRCKER, M.—*Investigation of the citrate and the molybdenum methods for phosphoric acid determinations in fertilizers.*

The paper is a report of the fertilizer committee of the Society of German Experiment Stations for 1892, in regard to methods of determination of phosphoric acid in fertilizers. The results obtained by thirty-five chemists in analyzing a sample of water-soluble P_2O_5 by the two methods are given in the report. Fifty grams of the solution sent out contained about 0.24 gram of $Mg_2P_2O_7$ or about 0.15 gram P_2O_5; the results found by the citrate method agreed closely, only three out of the thirty-five analyses varying more than 1.5 milligrams. The molybdenum method gave less satisfactory results, eleven chemists reporting analyses varying more than 1.5 milligrams (maximum 4.8 milligrams = very nearly 2 per cent). The main source of error in the latter method lies, according to the report, in precipitating the phosphoric acid with magnesia mixture in an ammoniacal (2.5 per cent) solution.

The greatest difference in the results found by the two methods was 2.4 grams P_2O_5, while those found by individual chemists ranged 3.6 milligrams of P_2O_5 according to the molybdenum method, and 3.5 milligrams with the citrate method. (For details as to methods of analysis, see original paper, pp. 102–106. Discussion of the report also published.)

Landw. Vers.-Stat. **41,** 329–373; **42,** 100–127. Jour. Chem. Soc., **64,** ii, 207, 208; Chem. Centralbl, 1892, i, 501; Exp. Sta. Record, **4,** No. 11. F. W. W.

E.—DIRECT MOLYBDATE METHODS.

12. BABBIT, H. C.—*Emmerton's process for the estimation of phosphoric acid.*

If the precipitation with molybdate is made at a temperature exceeding 25° the precipitate may contain arsenic. The yellow precipitate contains 1.63 per cent of phosphorus.

Jour. anal. Chem., **7,** 165–168; Chem. Centralbl., 1893, i, 957, 958; Jour. Chem. Soc., **64,** A, 553. J. W. F.

13. VILLIERS, A., and BORG, F., May, 1893.—*The determination of phosphoric acid (Sur le dosage de l'acide phosphorique).*

According to certain authorities the composition of precipitated ammonium phosphomolybdate is somewhat variable, and the estimation of phosphoric acid from the weight of the precipitate leads to uncertain and inaccurate results.

Still other authorities hold a contrary view, and the authors concluded, therefore, to determine the conditions under which correct estimations could be made directly by weighing the molybdic precipitate. The molybdate solution employed was that prescribed by Sonnenschein and Eggertz, and about 100 cc. were employed in the presence of 0.1 gram of phosphoric acid.

The phosphatic solutions used for the experiments were free from iron and alumina, and the precipitations were found to be best effected below 15° C., since, with a higher temperature, there was danger of the precipitate passing through the filter. The reagent was run into the precipitating vessel very carefully, and the liquid was not stirred until after two hours. The separation of the phosphomolybdate is found to be most complete when the solution, after precipitation in the cold, is kept at 40° C. for about four hours, but this temperature should not be exceeded for fear of precipitation of molybdic acid. The presence of potassium salts, it is found, does not occasion, as has been supposed, a change in the composition of the precipitate by a partial substitution of potassium for ammonium, and in consequence no error is caused thereby.

The precipitate is carefully washed, at first by decantation and then on the filter, employing water containing one-twentieth volume of molybdic solution; the filter and contents are weighed after careful drying for six hours at not over 100°.

The composition of the precipitate thus obtained corresponds to the formula $P_2O_5.24MoO_3.3(NH_4)_2O.3H_2O$, while the percentage proportion of P_2O_5 is 3.728.

The authors report results of tests with solutions of sodium phosphate alone, and also in the presence of salts of potassium, ammonium, lime, magnesia, manganese, zinc, etc., the best results being secured by maintaining the solution, at first two hours in the cold, and then four hours at 40° C.

Phosphoric acid can be readily determined in this manner in the absence of alumina, iron, silica, and such organic and other substances as exercise a reducing influence upon molybdic acid.

In the presence of iron and alumina, the phosphoric acid can be determined by dissolving the phosphomolybdate in ammonia, acidifying with tartaric acid, making slightly alkaline with ammonia, and precipitating with magnesia mixture.

This process is readily applicable to the determination of phosphoric acid in natural phosphates of lime.

Bull. Soc. Chim., **9** [3], 486-490; Compt. rend., **116,** 989; Jour. Chem. Soc., **64,** A, 434; Jour. Soc. Chem. Ind., **12,** 787; Ztschr. angew. Chem., 1893, 653; Ztschr. anorg. Chem., **5,** 250. B. B. R.

F.—TABLE OF PERCENTAGES.

14. SCHEIDING, FR., August, 1892.—*Table for calculation of phosphoric acid percentages (Tabelle zur Berechnung der Phosphorsäure bei Anwendung von 0.5 gram Substanz).*

This table gives the percentages of phosphoric acid based upon the employment of 0.5 gram of substance, for weights of magnesium pyrophosphate varying from 0.020 up to 0.350 gram. The percentages are given throughout the greater portion of the table for every 0.2 milligram, so that the correct figures can, in nearly every case, be taken directly from its columns. The factor used in making the calculations was 0.63976.

Chem. Ztg., **16,** 1145. B. B. R.

GENERAL VOLUMETRIC METHODS.

A.—URANIUM.

15. COLEMAN and GRANGER.—*The volumetric determination of calcium phosphate by means of uranium solution.*

Jour. Soc. Chem. Ind., **11,** 328; Ber. chem. Ges., **25,** 804; Vjschr. Chem. Nahr., **7,** 208.

B.—MALY METHOD.

16. GLÜCKSMANN, C.—*A volumetric determination of phosphoric acid (Zur quantitativen Bestimmung der Phosphorsäure).*

Gives direction for determining phosphoric acid by the Maly method.

Ztschr. allgem. Oesterr. Apotheker-Verein., 1892, 217; Chem. Centralbl., 1893, i, 663. E. W. A.

C.—LEAD NITRATE METHOD.

17. WAVELET, C.—*New process for the volumetric determination of phosphoric acid.*

The principle of the process depends upon the fact that in aqueous and acetic acid solutions a lead phosphate of constant composition is precipitated on addition of a soluble lead salt.

In the determinations the following solutions are necessary: (1) A solution of disodium phosphate (10.085 grams per liter); (2) a 5 per cent sodium acetate solution; (3) a 4 per cent lead nitrate solution; (4) a 5 per cent potassium iodid solution as an indicator. The titrations in standardizing are effected in the cold.

For the determination, 2 grams of superphosphate are employed and the solution made up to 200 cc. Fifty cubic centimeters of this are precipitated as magnesium-ammonium phosphate and the precipitate dissolved in dilute nitric acid. The solution is now made slightly alkaline with ammonia, and then acidified with a little dilute acetic acid, the volume being next made up to 200 cc. Fifty cubic centimeters of this solution, to which 5 cc. sodium acetate have been added, are employed for the titration.

With raw phosphates 5 grams of substance are weighed up, dissolved in dilute nitric acid, and the solution made up to 250 cc., of which 50 cc. are used in the determination.

Schweiz. Wochenbl. Pharm., **21,** 173, 174; Rep. Pharm., **49,** 153; Chem. Ztg. Rep., **17,** 122; Jour. Chem. Soc., **64,** A, 597. B. B. R.

D.—SILVER NITRATE METHOD.

18. HOLLEMANN, A. F., April 1893.—*Volumetric estimation of phosphoric acid.*

In a flask of 200 cc. capacity are placed 50 cc. of the liquid to be analyzed (containing less than 0.2 gram of phosphoric acid), 10 cc. of a normal solution of sodium acetate, and then a slight excess of decinormal silver nitrate. The solution is then neutralized with decinormal soda, using phenolphthalein as indicator. The phosphoric acid is, in the presence of sodium acetate, completely precipitated as silver phosphate, and the excess of silver is determined by diluting to 200 cc. and titrating 100 cc. with ammonium thiocyanate. Hydrochloric acid, ammonia and heavy metals must be absent.

Rec. Trav. Chim., **12,** 1-11. Jour. Chem. Soc. **64,** A, 490. Ber. chem. Ges., **26,** R, 728; **27,** R, 343. Ztschr. anal. Chem., **33,** 185. Agric. Science, **8,** 137, 138. Chem. Ztg., **17,** 545; **18,** Repert., 545. Chem. Centralbl., 1894, i, 1012. J. W. F.

E.—FERRIC AMMONIUM ALUM METHOD.

19. SPICO, MATTEO.—*A volumetric method for the estimation of* P_2O_5.

After obtaining the phosphate in solution by use of absolute alcohol, drying and taking up with water (Fe, Al, and Mn salts must be absent), the author neutralizes exactly with caustic alkali, using phenolphthalein as indicator; he then adds a small quantity of salicylic acid, and titrates with a solution of ferric ammonia-alum (1 cc. =0.001 gram P_2O_5, previously standardized with a solution containing 2.9439 grams microcosmic salt per liter, of which 1 cc. also =0.001 gram P_2O_5), adding it in small quantities at a time. Test experiments very satisfactory.

Chem. Ztg., **16,** Repert., 141; Vjschr. Chem. Nahr., **7,** 207; Gazz. Chim. Ital., **22** (1), 117; Analyst, **17,** 116, 117; Jour. Chem. Soc., **62,** 912. F. W. W.

20. ARNOLD, C., and WEDEMEYER, K., 1892.—*Spico's process for the estimation of phosphoric acid.*

The authors have investigated the process, but can not recommend it, as alcohol is not a satisfactory separant, and results are too low in consequence.

Ztschr. angew. Chem., 1892, 603, 604; Jour. Chem. Soc., **64,** ii, 299; Ztschr. angew. Chem., 1892, 603; Jour. anal. Chem., **6,** 598; Chem. Centralbl., 1892, ii, 938.

J. W. F.

F.—COLORIMETRIC METHOD.

21. NASTJUKOW, A.—*Colorimetric method for the estimation of phosphoric acid in phosphorites.*

When phosphorites are ignited the color changes to a dark-blue gray, owing to the formation of ferrous phosphate. The same color can also be obtained by igniting phosphate of lime with oxid of iron and carbon. The author's experiments on the ignition of artificial mixtures of tricalcium phosphate, ferric oxid, albumin and calcium carbonate, in different proportions, show that the intensity of this color is dependent on the proportion of phosphoric acid present. The test is characteristic of phosphorites and is not obtained when marls, limestones, sandstones, etc., are ignited. On these facts the author basis a quantitative colorimetric test, which is accurate within 2 to 5 per cent when the phosphoric acid present is not less than 6 to 8 per cent and not more than 30 per cent.

Jour. Russ. Chem. Soc., **125,** 31-42; Ber. chem. Ges., **26,** Ref., 390; Jour. Chem. Soc., **64,** A, 553. A. L. W.

G.—ALKALIMETRIC METHOD.

22. HUNDESHAGEN, F., 1894.—*Volumetric determination of magnesia and phosphoric acid by titrating the ammonium-magnesium phosphate or the alkaline earths in the form of triphosphate (Volumetrische Bestimmung der Magnesia, der Phosphorsäure und der Arsensäure durch Titration des Magnesium-Ammonium-Phosphats bezw. Arseniats, sowie der alkalischen Erden in Form der Tripelphosphate).*

The precipitate of magnesium-ammonium phosphate or arsenate, obtained in the usual way, as soon as the separation is complete, is transferred to a strong filter or perforated Gooch crucible. The beaker, precipitate, and filter are washed with 2.5

per cent ammonia solution until free of the adhering liquid; the ammonia is removed by alcohol. To secure the precipitation to the best advantage, no more substance or solution is used than is equivalent to about 0.1 gram magnesium oxid (equals 50 cc. $\frac{N}{10}$ acid.) Precipitate in a beaker of not more than 250 cc.content and select a filter of from 5 to 9 cc. diameter, according to the mass of the precipitate. In washing the precipitate, about 15 to 30 cc. of ammonia solution are used, and about the same amount of alcohol. Wash the precipitate from the filter or crucible into a beaker. Add from 5 to 15 drops of cochineal solution; then run in acid from a burette, at first in large portions, then drop by drop, until the precipitate is dissolved and the alkaline reaction disappears. The acid used can be either hydrochloric, nitric, or sulphuric $\left(\frac{N}{10}\right)$.

Chem. Ztg., **18**, 445, 446; Chem. Centralbl., 1894, i, 876, 877. J. P. S.

H.—MOLYBDATE METHOD.

23. PEMBERTON, H., JR., 1893.—*The determination of phosphoric acid by the titration of the yellow precipitate with standard alkali.*

The author calls attention to a process suggested by him in 1882, where the amount of yellow precipitate (ammonium-phosphomolybdate) was determined by means of a standard solution of caustic alkali, using litmus as an indicator. He has applied it to samples of various composition, and finds it in every case very satisfactory.

Solutions: Ammonium molybdate.—Ninety grams of the crystals are dissolved in somewhat less than one liter of water. This is allowed to settle over night and the clear liquid decanted into a liter flask. The small amount of insoluble molybdic acid is dissolved in a little ammonia and added to the main solution. The whole is made up to one liter. It is this *aqueous* solution that is used, no nitric acid whatever being employed. Each cubic centimeter precipitates 3 milligrams of P_2O_5.

The *ammonium nitrate* solution is a saturated, aqueous solution of the salt.

The *nitric acid* used has a specific gravity of 1.4.

The *standard potassium hydroxid* solution is of such strength that 1 cc. equals 1 milligram P_2O_5; 100 cc. of it will neutralize 32.65 cc. of normal acid.

The *standard acid* has the same strength, volume for volume, as the potassium hydroxid, and can be made by diluting 326.5 cc. of normal acid to 1 liter.

The *indicator* can be either litmus, rosolic acid, or phenolphthalein. The author used the latter almost exclusively, finding that it is reliable in the presence of ammonium salts if the amount of the salt present is not excessive, if the solution is cold, and if the phenolphthalein is used in sufficient quantity. One gram of the phenolphthalein is accordingly dissolved in 100 cc. of 60 per cent alcohol, and at least 0.5 cc. of this solution is used for each titration.

The method is as follows: One gram of phosphate rock or 2 or 3 grams of fertilizer are dissolved in nitric acid, and without evaporating to dryness, diluted to 250 cc. The solution need not be filtered. Twenty-five cubic centimeters of the solution are delivered into a 4-ounce beaker, and neutralized with ammonia until a precipitate just begins to form, and then treated with 5 cc. of nitric acid of 1.4 sp. gr. Ten cubic centimeters of a saturated solution of ammonium nitrate are added and the solution diluted to a volume of from 50 to 75 cc. It is then brought to a full boil, removed from the lamp, and 5 cc. of the aqueous solution of ammonium molybdate added. This is followed by a second and a third 5 cc., if necessary, the precipitate allowed to settle, and filtered at once through a 7-centimeter filter. It is washed thoroughly with water by decantation and on the filter. The filter and precipitate are transferred bodily to the beaker. Standard alkali is then run in and at least 0.5 cc. of phenolphthalein solution added. and then standard acid until the color vanishes. Each cubic centimeter of alkali equals 1 milligram of phosphorus pentoxid.

Jour. Am. Chem. Soc., **15**, 382–395; Jour. Frank. Inst., **136**, 362–376; Chem. Centralbl., 1894, i, 105; Chem. Ztg., **17**, Repert., 318; Jour. Soc. Chem. Ind., **13**, 176; Exp. Sta. Record, **5**, 444; Agric. Science, **8**, 26–31. J. P. S.

24. PEMBERTON, H., JR., 1894.—*On the determination of phosphoric acid.*

As a result of further tests, the author finds that the ratio of the phosphorus pentoxid of the precipitate and the standard alkali was 1:23, instead of 1:23.2, as previously reported. The standard acid is, therefore, prepared by diluting 323.7 cc. of normal sulphuric acid to 1 liter, and not 326.5, as previously stated.

Jour. Am. Chem. Soc., **16**, 278–282; Jour. Frank. Inst., **137**, 304, 305; Chem. Centralbl., 1894, i, 927; Agric. Science, **8**, 90. J. P. S.

25. DAY, W. C., and BRYANT, A. P., 1894.—*Note on Pemberton's method of phosphoric acid determination as compared with the Official methods.*

The authors have compared the Pemberton with the Official methods and obtained very satisfactory results. They would recommend it, because of its extreme accuracy, easy manipulation, and rapidity. They find that from thirty to forty minutes for a single determination, from the time the solution is measured out for titration, is quite sufficient.

Jour. Am. Chem. Soc., **16**, 282, 283; Jour. Frank. Inst., **137**, 394, 395; Chem. Centralbl., 1894, i, 927, 928; Agric. Science, **8**, 134. J. P. S.

26. WILLIAMS, W. J., 1894.—*Comments on the determination of phosphoric acid by titration of the yellow precipitate, as described by Mr. Henry Pemberton, jr.*

The Pemberton method, after a number of tests, was found entirely reliable for high-grade rocks, for plain acid phosphates, and for mixed fertilizers in the hands of careful and competent analysts. With careless or unskillful manipulation the chances of serious error are very great.

Jour. Frank. Inst., **137**, 126–128.

27. BERGAMI, F., 1894.—*Comparison of Pemberton's method of phosphoric acid determination with the Official method.*

Extremely great care must be taken in washing the yellow precipitate until free from acid. Never omit to test with delicate litmus paper. For the sake of rapidity, the author suggests a few changes in Pemberton's mode of procedure.

To the 25 cc. of acid solution add at once 10 cc. of strong ammonia (sp. gr. 0.90) and after a little stirring 15 cc. of nitric acid (sp. gr. 1.42). This operation secures about the same conditions for all tests and produces at the same time a temperature of 75° to 80° C., which reduces the time for heating to boiling point to a minimum. He found that it was unnecessary to have the standard solution absolutely free from carbonic acid, provided phenolphthalein and cochineal are used as indicators for standardization and analysis, respectively. He found a little inconvenience in such samples as gave dark-colored acid solutions, thus rendering the titration difficult. A previous ignition of the sample or treating it with concentrated sulphuric acid would overcome this difficulty. Slightly lower results were obtained than by the official method, doubtless due to the difficulty experienced in obtaining a precipitate of ammonium-magnesium phosphate absolutely free from magnesium oxid.

Jour. Frank. Inst., 1894, **137**, 129, 133. J. P. S.

TREATMENT OF SPECIAL PHOSPHATIC SUBSTANCES.

A.—FERTILIZERS RICH IN ORGANIC MATTER.

28. WEIBULL, M., November, 1892.—*Analysis of fish guano, bone meal, poudrette, and like substances (Zur Analyse von Fischguano, Poudrette, Knochenmehl, und dergl).*

Reference is made to the difficulties attendant upon the determination of phosphoric acid in substances containing large proportions of organic matter. The usual methods, such as ignition, fusion with Na_2CO_3 and KNO_3, solution in aqua regia or in HCl with $KClO_3$ are pronounced unsatisfactory in certain particulars, and in their place the Kjeldahl method is recommended, employing cupric oxid as an oxidizing agent. Results of determination of phosphoric acid in pure ammonium phosphate are given, employing both 5 and 10 cc. of sulphuric acid, and the figures obtained agree quite well with the theoretical percentages. Tests were also made upon fine and coarse bone meal, poudrette, and fish guano, using both the Kjeldahl and the usual methods of analysis, the results obtained being quite concordant.

Two and five-tenths grams of substance in the process used by the author are digested with 20 cc. concentrated H_2SO_4, after addition of a little cupric oxid; the solution is made up to 250 cc., and after filtration 50 cc. of the liquid are treated as in the regular method.

[NOTE BY ABSTRACTOR.—The author makes no reference to the method of de Roode, already successfully employed for several years.]

Chem. Ztg., **16,** 1689; Chem. Centralbl., 1893, i, 131. B. B. R.

29. WEIBULL, M., 1894.—*Further contribution to the analysis of fish guano, poudrette, and similar substances (Weitere Beiträge zur Analyse von Fischguano, Poudrette, und dergl. Substanzen).*

In line with the author's previous recommendation of the application of the Kjeldahl method to the simultaneous determination of phosphoric acid and nitrogen in bone meal, fish guano, and similar substances, he now extends the method to the determination of lime in cheese, milk, and other foods, the amount of substance taken for analysis, of course, being increased. In the analysis of peat dust poudrette, the following method was used: 100 grams of material were ground up with 400 grams of concentrated sulphuric acid; 50 grams of the mixture were taken, mixed with copper oxid, and treated as usual by the Kjeldahl method. At the end of the digestion the solution was washed into a 250 cc. flask with water, and the flask filled to the mark; 100 cc. of this solution served for the determination of phosphoric acid and a like amount for that of lime; in the remainder nitrogen was determined. Only in the case of the determination of phosphoric acid by the citrate method was filtering of the solution necessary.

To determine the lime the solution was exactly neutralized with ammonia and a few drops of hydrochloric acid added until the reaction was slightly acid. To the hot solution sufficient ammonium oxalate was added, and the mixture allowed to stand two hours at the temperature of the water bath. The determination of the calcium oxalate by titration with permanganate, according to Immendorf, is recommended. Careful tests of this method, under varying conditions and comparisons with other methods, indicated that it was as reliable as those ordinarily employed. It possesses the further advantages of rapidity and simplicity of manipulation.

Chem. Ztg., **18,** 31, 32; Exp. Sta. Record, **5,** 802. J. P. S.

30. HESS, W.—*The sampling of ground bone and the determination of nitrogen and phosphoric acid in the same weighed portion (Ueber die Untersuchung von Knochenmehl, Fleischmehl, und ähnlichen phosphorsäure- und stickstoffhaltigen Düngemitteln).*

The author finds that the discrepancies in results obtained on the same lot of ground bone in different laboratories are due oftentimes to errors in sampling rather than in the analytical work. As ground bone generally consists of a mixture of material of different fineness, specific gravity, and composition it is difficult to secure the thorough admixture necessary for a good analysis. The determination of nitrogen and phosphoric acid in aliquots from one, rather large (5 grams), weighed portion tends to diminish the error, and for this purpose the author describes a method essentially the same as that described by de Roode on page 104 of the proceedings of this association for 1891.

Ztschr. angew. Chem., 1893, 74; Chem. Centralbl., 1893, i, 583; Jour. Chem. Soc., **64,** A, 389. A. L. W.

31. OTTO, H., August, 1892.—*The solubility of the phosphoric acid of bone meal* (*Die Löslichkeit der Phosphorsäure des Knochenmehls*).

The result of a number of experiments with regard to the solubility of the phosphoric acid of bone meal are reported, material of different degrees of fineness being employed in making the tests. Wagner's citrate solution was used and the original method of Wagner was followed in conducting the experiments. By using material passed through a sieve with a 1-mm. mesh, quite perceptibly higher results were obtained than where a 1½-mm. mesh was employed. The phosphoric acid in fat-free bone meal was found to be more readily soluble than that in the ordinary article.

Chem. Ztg., **16**, 1128; Exp. Sta. Record, **4**, 378. B. B. R.

32. DANCY, F. B.—*Estimation of phosphoric acid in fertilizers containing cotton-seed meal.*

The solution of such samples in nitric acid with a small quantity of hydrochloric acid does not give up all of its phosphoric acid to molybdate, and the same is true of the solution with hydrochloric acid and potassium chlorate. Fusion with equal parts of sodium carbonate and potassium nitrate gives maximum results, but takes too long, and incineration, and solution in nitric acid gives low results owing to the insolubility of the ignited phosphate. Hydrochloric acid was found to dissolve the residue easily and completely, thus furnishing a satisfactory basis for a desirable method.

Chem. News, **65**, 162–163, 170–172; Jour. Chem. Soc., **62**, 1029. J. W. F.

B.—BASIC SLAG.—(1) SOLVENT FOR.

33. FOERSTER, OTTO, October, 1892.—*Estimation of value of Thomas slag* (*Zur Werthbestimmung der Thomasschlacken*).

A comparison is made of the different methods for effecting solution of Thomas slag, the advantages and disadvantages attendant upon the use of each solvent being set forth. By employment of nitric acid, or nitrohydrochloric acid, the phosphorus combined with the iron is oxidized to phosphoric acid, while with the use of hydrochloric acid, the magnesium ammonium phosphate precipitate is contaminated with various impurities which are brought into solution, these impurities in some cases amounting to as much as 6.33 per cent of its weight.

With concentrated sulphuric acid as a solvent the iron phosphid is also oxidized and a considerable error is occasioned on account of the amount of gypsum formed; the sulphuric acid also dissolves less phosphoric acid than the hydrochloric. Dilute sulphuric acid, however, is without the oxidizing action of the concentrated acid, and solution can be effected by boiling only a short time.

The author thinks that since calcium tetraphosphate is perfectly soluble in dilute citric-acid solution, a method should be devised which depends upon the solubility of this phosphate in a citric acid or ammonium citrate solution.

Chem. Ztg., **16**, 1597; Chem. Centralbl., 1892, ii, 938. B. B. R.

34. FOERSTER, OTTO.—*Valuation of basic slag.*

The best results are given by boiling the sample with dilute sulphuric acid and using the molybdate method.

Chem. Ztg., **16**, 1596, 1597; Jour. Chem. Soc., **64**, A, 344. J. W. F.

35. JOLLES, A. F., October, 1892.—*The determination of phosphoric acid in Thomas slag* (*Ueber die Phosphorsäurebestimmung in Thomasschlacken*).

By the employment of sulphuric acid as a solvent in the analysis of Thomas slag, the author has been unable to secure correct results, by either following the molybdate or the citrate processes for the determination of phosporic acid, the source of the errors lying chiefly in the fact that the calcium sulphate formed incloses particles of calcium phosphate, thus protecting them from the action of the solvent.

The method of H. and E. Albert, employing hydrochloric acid to dissolve the slag and boiling down to a jelly-like mass, with subsequent dilution with water, yields in the hands of the author entirely too high results, due, in his opinion, to the incomplete elimination of the silica.

The following modification of Albert's method is therefore recommended: 5 grams of the finely divided slag are moistened with water in a dish, boiled with 40 cc. of hydrochloric acid (about 20° B.) and evaporated to complete dryness.

The residue is heated in an air bath to 120° C., moistened with hydrochloric acid after cooling, taken up with water and filtered, the filtrate being made up to 500 cc. Fifty cubic centimeters of this are mixed with citrate solution (Joulie's), precipitated with magnesia mixture and the precipitate dried, ignited, and weighed in usual manner. A number of results of determinations by means of this method, the original process of Albert and the molybdic method are given, the figures by the first and last named processes being quite concordant.

Ztschr. anal. Chem., **31**, 516–519; Ztschr. angew. Chem., 1892, 737; Jour. anal. Chem., **6**, 625; Jour. Chem. Soc., **64**, ii, 88–89. B. B. R.

36. FIRBY, A., December, 1892.—*The determination of phosphoric acid in basic slags.*

Referring to Jolles's article on the determination of phosphoric acid in basic slags, the author concurs in the conclusions of the former with regard to the inadvisability of employing sulphuric acid as a solvent, and also with reference to the incomplete separation of silica by the Albert method.

The author had previously made experiments leading to the same results as those of Jolles, and is surprised that chemists of experience should think of employing sulphuric acid in dissolving these slags. In separating silica completely, two evaporations have been frequently found necessary, and the recommendations of Jolles on this point can be advantageously followed. The Joulie gravimetric method is considered to be untrustworthy for accurate determinations, the results secured being almost invariably too high.

Chem. News, **66**, 293. B. B. R.

37. MÄRCKER, M.—*Determination of phosphoric acid in Thomas slag.*

A part of the preceding report (No. 11); the boiling with HCl and with H_2SO_4, preliminary to the phosphoric acid determination investigated; the result of the work done showed that the slag ought to be boiled with H_2SO_4. The method recommended is to heat 10 grams of slag in a dry 500 cc. flask with 50 cc. conc. H_2SO_4 over free flame, under constant shaking until boiling. After standing a little, water is carefully added, and when cold filled to the mark and filtered; 50 cc. taken for P_2O_5 determination by the citrate method.

Jour. Chem. Soc., **64**, ii, 237, 238; Chem. Centralbl., 1893, i, 502; Exp. Sta. Record, **4**, No. 11; Landw. Vers.-Stat., **41**, 371–373; **42**, 134, 135. F. W. W.

38. MANCUSO-LIMA, G.—*Estimation of phosphoric anhydrid in basic slag.*

Two grams of the powdered slag are treated in a flat dish with 25 cc. of sulphuric acid diluted with half its volume of water, stirring rapidly with a glass rod. The pasty mass thus formed is left for about two hours, treated with warm water, filtered and washed. Milk of lime free from phosphates is then added, the whole shaken, and when alkaline, heated for half an hour on the water bath, treated with nitric acid until the lime precipitate is redissolved, allowed to cool, and filtered. The solution is precipitated with molybdate and magnesia mixture as usual.

Staz. sper. agr. Ital., **21**, 225–228; Jour. Chem., Soc., **64**, ii, 238. J. W. F.

39. MARTELLI, D.—*Estimation of phosphoric anhydrid in basic slag.*

The author has compared the methods of Martinotti (Staz. Sper. Agr., **19**, 614), Olivieri (*ibid.*, **20**, 159), and Mancuso-Lima (*ibid.*, **21**, 225 and 453), with that given by König in his Untersuchung landw.- und Gewerbewichtiger Stoffe (Berlin, 1891, pp.

163, 178, 179). He considers the methods described by König to be the best, as effecting a saving both of time and reagents.

Staz. sper. agr. Ital., **19**, 614; **20**, 159; **21**, 225, 453; Jour. Chem. Soc., **64**, ii, 238, 239; Analyst, **17**, 116. J. W. F.

B.—BASIC SLAG (CONTINUED).—(II). ESTIMATION OF LIME IN.

40. HOLLEMAN, A. F., October, 1892.—*Lime determination in Thomas phosphate* (*Methode zur Kalkbestimmung in Thomasphosphaten*).

The chief difficulty in the way of the accurate determination of lime in Thomas phosphate lies in the fact that by the methods ordinarily employed the lime precipitate is contaminated, invariably, with iron and manganese. Both the method of Classen, using potassium oxalate, and that of Jones, precipitating as sulphate in an alcoholic solution, failed to give satisfactory results, though the latter process was the more accurate of the two. In order to arrive at a method adapted to work of this character, a series of experiments were made both upon Thomas slag and a prepared mixture of known composition containing all the chief constituents of the former. The results indicated that by combining the methods of Classen and Jones the iron and manganese could be completely eliminated.

The solution of material is effected by means of hydrochloric acid, an aliquot part of the filtered solution being employed for the determination. This is concentrated to small bulk, solution of neutral potassium oxalate is added, and the mixture digested on water bath for about ten minutes. The precipitate is brought on filter, well washed with hot water, and dissolved in hot dilute HCl. Precipitation is now effected with dilute sulphuric acid in the presence of 96 per cent alcohol, as in the Jones method, the calcium being finally weighed as sulphate.

Maandbl. voor Natuurwetensch, 18, 1–4; Chem. Ztg., 16, 1471; Chem. Centralbl., 1893, i, 796. B. B. R.

B.—BASIC SLAG (CONTINUED).—(III). DETECTION OF ADULTERANTS.

41. DYER, B.—*The adulteration of basic slag.*

The difference in solubility in 5 per cent citric acid solution of phosphoric acid of the slag and that of Redonda phosphate is of no value in detecting the latter adulterant of slag, according to the author, as calcined Redonda phosphate is quite soluble in the solution, and this phosphate is apt to be used in adulterating the slag.

Analyst **17**, 4. F. W. W.

42. O. BÖTTCHER, 1894.—*The detection of adulterations of Thomas meal* (*Zum Nachweis von Verfälschungen des Thomasmehles*).

There are certain fluorin compounds in phosphorites which are not in Thomas meal. Ten to 15 grams of the suspected meal are placed in a beaker 10 cm. in height and from 5 to 6 cm. in width. This is saturated with 15 cc. of concentrated sulphuric acid, stirred around with a glass rod, and the beaker covered with a watch glass, from which hangs a drop of water. If a dirty white color forms around the edge of the drop, it indicates that phosphorites have been added. After a few minutes the etching caused by the hydrofluoric acid can be clearly seen on the cleaned watch glass. An addition of 10 per cent raw phosphate can be detected in this way.

Chem. Ztg., **18**, 565, Chem. Centralbl., 1894; i, 1102. J. P. S.

C.—IRON AND ALUMINA PHOSPHATES.

43. LADROWICZ, B.—*Action of water and of saline solutions upon ferric phosphates.*

The author made observations interesting to the analyst, that pure water, solutions of neutral salts of alkalies and especially solutions of alkali salts of alkaline reaction, partially dissociate ferric phosphate, removing phosphoric acid, but not the iron oxid. Salts of acid reaction, such as ferric chlorid, also decompose ferric phosphate, but remove both phosphoric acid and iron oxid. Free humic acid has no action, but alkaline humate without excess of alkali dissolves somewhat more of both phosphoric acid and iron oxid than would have dissolved in an equivalent quantity of the alkali acting alone.

Monatsh. f. Chem., **13,** 357; Bull. Soc. Chim [3], **8,** 1218. W. F.

44. BORNTRÄGER, H., March, 1893.—*Simple separation of ferric oxid and alumina (Einfache Trennung des Eisens von der Thonerde).*

In determination of iron and alumina, especially in fertilizer analysis, it is customary to precipitate them with ammonia, and their separation is somewhat difficult, according to the methods heretofore followed. The mixed oxids of iron and aluminum, after ignition and weighing, are dissolved in hydrochloric acid, and potassium hydrate solution is added to neutrality. The precipitation of both oxids is effected in the hot solution by means of neutral potash soap solution. The precipitate, after filtration and washing with hot water, is dried for awhile over calcium chlorid. By treating the gelatinous mass with hot petroleum until the precipitate loses its color the ferric oleate is dissolved out, leaving the aluminum oleate, which, on ignition, leaves alumina. The petroleum solution of ferric oleate is ignited in platinum dish and the residue, after completion of ignition, is weighed as ferric oxid.

Ztschr. anal. Chem., **32,** 187; Jour. Chem. Soc., **64,** ii, 304; Jour. anal. Chem., **7,** 235. B. B. R.

45. GLASER, C., July, 1892.—*Estimation of aluminum phosphate (Ueber die Bestimmung von Thonerdephosphat).*

The following modification of the usual method is recommended: To the phosphate solution, which must not contain free chlorin, a drop of methyl-orange solution is added, and then ammonia, until the acid is all but neutralized. A few cubic centimeters of ammonium acetate now change the color to yellow. The mixture is then heated to 70° C., when the iron and aluminum phosphates are completely precipitated. If calcium phosphate is present a little of it adheres to the precipitate, which should be redissolved in hydrochloric acid and precipitated as before, after adding a little sodium phosphate. Should the original liquid contain free chlorin, ammonia and then hydrochloric acid must be added before the indicator. The precipitate is washed, using the filter pump, gently ignited, and weighed. To estimate the iron in the precipitate it is covered with pure sodium carbonate and fused for 10 minutes, using the blowpipe. The fused mass is boiled with water and filtered hot, and the ferric oxid washed well with boiling water. Hot filtration is necessary to prevent the separation of an aluminum phosphate of the composition $Al_3P_2O_7$, which is sparingly soluble in cold soda solution. The iron oxid is dissolved and reprecipitated before weighing.

Ztschr. anal. Chem., **31,** 383–388; Pharm. Rev., 1892, Oct.; Jour. Chem. Soc., **62,** 1523, 1524; Jour. anal. Chem., **6,** 527. J. W. F.

46. GLASER, C.—*Separation of iron oxid from alumina in phosphatic rocks by fusion with an alkali carbonate.*

The author suggests the following as a modified method giving more accurate results than the method as at first published: The hydrochloric acid solution is nearly neutralized with ammonia, using methyl-orange as an indicator. Ammonium acetate is then added until the color changes to a pale yellow. The mixture is then heated on the water bath below 70°, thus separating the iron and aluminum phosphates completely. Filter and thoroughly wash with water below 70°. It is advisable to dissolve the precipitate, add a little sodium phosphate, and reprecipitate to remove calcium salts. Heat in a platinum crucible, being careful not to fuse the precipitate, and weigh. Then fuse with sodium carbonate, dissolve the fusion in hot water, filter, and wash the precipitate of ferric oxid with boiling water. Dissolve in hydrochloric acid, precipitate with ammonium hydrate, and weigh. Add HCl to the filtrate, and precipitate the aluminum as phosphate by adding ammonium acetate.

Jour. anal. Chem., **6,** 624–626; Jour. Chem. Soc., **64,** A, 346. J. W. F.

47. VOGEL, J. H.—*Analysis of phosphates.*

The author precipitates the aluminum and ferric phosphates according to Glaser's directions, but does not boil the solution. The phosphoric acid is now separated according to Stutzer's plan, with molybdate and precipitation of the ferric and aluminum hydrates with ammonia, leaving the magnesium in solution. A second precipitation frees the precipitate from any molybdate.

Chem. Ztg., **15**, 495–499; Jour. Chem. Soc., **62**, 912. J. W. F.

48. MARIANI, G., and TASSELLI, E.—*On the determination of ferric oxid and alumina in mineral phosphates.*

Staz. sper. agr. Ital., **23**, 31; Analyst, **18**, 90, 91. F. W. W.

D.—FREE PHOSPHORIC ACID.

49. WAGNER, J.—*Estimation of free acids in the presence of acid phosphates.*

Leo's method (Jour. Chem. Soc., 1891, 1288) is again criticised, and declared to be wholly worthless.

Pflüger's Archiv, **50**, 375–384; Jour. Chem. Soc., **64**, ii, 41. J. W. F.

E.—PYRO AND META PHOSPHORIC ACID.

50. VON KNORRE, G.—*The separation and determination of pyro and meta phosphoric acid.*

Ztschr. angew. Chem., 1892, 639; Chem. Centralbl., 1892, ii, 1046.

POTASH—GENERAL.

51. MUELLER, K.—*Examination of potassic fertilizers.*

Report of the committee on potash of the Association of German Experiment Stations. Discussion of methods of analysis and recommendation of continued investigation of the subject.

Landw. Vers.-Stat., **42**, 171–173. F. W. W.

52. ROBINSON, N. 1894.—*Some sources of error in our methods of determining potash in fertilizers and German potash salts.*

The writer, after a series of tests, finds that there are very noticeable losses of potash in the Lindo-Gladding, the alternate, and the Stassfurt methods.

The chief sources of error arise from the occlusion of potash salts by precipitates in all three methods, from loss in the alcohol washings, and through the solvent action of the ammonium chlorid solution in the Lindo-Gladding method. In a series of determinations the writer finds an average loss to each per cent of potash estimated by the Lindo-Gladding method of 0.029 per cent, and by the Alternate method a loss of 0.29 per cent.

Jour. Am. Chem. Soc., **16**, 364–372; Cf. Proc. A. O. A. C., 1893, Bull. 38, Chemical Div., U. S. Dept. of Agr. J. P. S.

A.—PLATINUM METHODS.

1. Solubility of salts.

53. PÉLIGOT, M.—*Solubility of potassium and sodium platinic chlorid in alcohol.*

One liter each of 50, 90, and 95 per cent alcohol dissolves respectively 0.6, 0.1, 0.03 gram of the potassium salt. The addition of one-fifth volume of ether to 95 per cent alcohol diminishes the amount dissolved to 0.027 gram. One liter of methyl alcohol dissolves 0.072 gram K_2PtCl_6. One liter of 60 per cent alcohol dissolves 110 grams Na_2PtCl_6, and the same quantity of 95 per cent alcohol dissolves 22 grams.

Monit. Scient., 1892 [4], **6**, 872; Ber. chem. Ges., **26**, Ref. 1051. A. L. W.

2. Lindo-Gladding method.

54. Breyer and Schweitzer.—*The determination of potash by the method of Lindão, as modified by Gladding.*

The authors attempt to prove, by quotations, chiefly from the bulletins of the U. S. Department of Agriculture, that the Lindo-Gladding method is inaccurate. From their own experiments they arrive at the following conclusions :

1. The addition of sodium chlorid is unscientific and injurious to the exactness of the results. The wash alcohol generally contained potash.
2. It is doubtful whether the method is a simplification of that of Fresenius.
3. By washing the precipitate with half-saturated ammonium chlorid solution a partial decomposition takes place, potassium chlorid being washed away, while ammonium chloro-platinate remains on the filter.
4. The removal of impurities from the precipitate is not complete, especially in the presence of sulphates of magnesium and calcium.
5. Results are sometimes too high, sometimes too low.

Jour. anal. Chem., **6,** 470 ; Ztschr. angew. Chem., 1893, 122. A. L. W.

55. Breyer and Schweitzer ; November, 1892.—*Defects of the Lindo-Gladding method (Falsche, aber officielle handelsanalytische methoden in den Vereinigten Staaten.)*

In substance, similar to the foregoing.

Chem. Ztg., **16,** 1720; Exp. Sta. Record, **4,** 584.

56. Breyer, T., and Schweitzer, H., January, 1892.—*The Lindo-Gladding potash method (Kalibestimmung nach der Methode Lindo-Gladding.)*

The report of the German kali works is quoted from, in which results of analyses are given which, the authors claim. show the unreliability of this method, thus substantiating the conclusions arrived at by them and reported in a previous article.

Chem. Ztg., **17,** 101; Ber. chem. Ges., **26,** Ref. 337; Chem. Centralbl., 893, i, 442; Jour. Chem. Soc., **64,** A, 436. B. B. R.

57. Breyer, T., and Schweitzer, H., February, 1893.—*The Lindo-Gladding potash method (Lindo-Gladding Methode zur Kalibestimmung).*

The authors, in reply to the article of Dr. Holleman (Chem. Ztg., **16,** 1920), reiterate some of their former statements and conclusions, though nothing essentially new is presented.

Chem. Ztg., **17,** 244; Chem. Centralbl., 1893, i, 585. B. B. R.

58. Holleman, A. F., December, 1892.—*Potash determination by the Lindo-Gladding method (Die Kalibestimmung nach der Methode Lindo-Gladding).*

The article of Breyer and Schweitzer with regard to this subject is referred to, and issue is taken with them with reference to their claims as to the unreliability of this method, since the results secured by the author have been uniformly satisfactory.

The results of the analysis of eight samples of kainit are reported, the potash determinations being made both by the Lindo-Gladding and the official Dutch method (Fresenius), there being quite a close agreement in the figures obtained by the two methods. For two of the samples the amounts of alcohol required for the first washing were 46 and 50 cc., respectively; of ammonium chlorid, 25 cc., for each sample, and 25 and 23 cc. of alcohol for the second washing. The potassium-platinum chlorid precipitates were found to contain not a trace of ammonia; a microscopic examination showed them to be pure, and tests showed only the very slightest trace of sulphuric acid, and hence no tests were made for presence of lime and magnesia. The presence of ammonium-platinum chlorid in the precipitates of Breyer and Schweitzer are attributed to incomplete washing out of platinic chlorid. The conclusions of the author are:

(1) The Lindo-Gladding method gives by proper execution correct results.

(2) There is no occasion for its abandonment.

(3) The experiments of Breyer and Schweitzer are not suited to establish the unreliability of the method.

Chem. Ztg., **16**, 1920; Chem. Centralbl., 1893, i, 319; Exp. Sta. Record, **4**, 586; Jour. Chem. Soc., **64**, A, 344. B. B. R.

3. Reduction methods.

I.—PURE SALT.—SPONGY PLATINUM.

59. HILGARD, E. W., March, 1893.—*The determination of potassium* (*Zur Bestimmung des Kaliums*).

The weighing of potassium-platinic chlorid gives in experienced hands very good results, especially if the Gooch crucible is employed. The process frequently used, however, yields, in many cases, erroneous results, and the author therefore proposed a method which, with care, gives very accurate figures. The potassium-platinic chlorid is reduced in a crucible, the lower half of whose interior is covered with a film of platinum sponge, formed by the previous ignition of about a decigram of double salt. In the presence of this sponge, the decomposition is effected readily even at a comparatively low heat, though the crucible is ignited strongly at the last. About two drops of HCl are added to the crucible when cool, and if, after gentle warming, a yellow color appears, a little oxalic acid is added and the ignition continued. By washing with acidified water, the soluble substances present are removed, among which are frequently traces of chlorid of sodium, earths, etc. On this account, this process gives more exact results than the weighing as double salt.

Ztschr. anal. Chem., **32**, 184; Jour. Chem. Soc., **64**, ii, 300, 301; Rev. internat. falsificat., 745; Chem. Centralbl., 1893, i, 795; Ber. chem. Ges., **26**, R, 506.

B. B. R.

I.—PURE SALT (CONTINUED).—SOAP METHOD.

60. BORNTRÄGER, H., March, 1893.—*Quick reduction of potassium-platinic chlorid* (*Rasche Reduction des Kaliumplatinchlorids*).

The simplest and quickest way to reduce potassium-platinic chlorid is as follows: The double salt is dissolved in boiling water and the platinum is precipitated with a potash soap solution. The precipitate having settled, the supernatant liquid is poured off and the residue, which consists of a double salt of potassium chlorid and platinum oleate, is ignited in a porcelain crucible. After heating a few minutes the platinum black which is obtained can be washed free from potassium chlorid with hot water.

Ztschr. anal. Chem., **32**, 188. B. B. R.

I.—PURE SALT (CONTINUED).—FORMALDEHYDE METHOD.

61. JEAN, F., and TRILLAT.—*Estimation of potassium.*

The platinochlorid, having been precipitated and purified in the usual way, is dissolved in water, and the solution, made alkaline with soda, is warmed and reduced by the addition of a few drops of aqueous formaldehyde. It should be stirred with a glass rod to prevent the platinum adhering to the vessel.

Bull. Soc. Chem., **7** [3], 228; Jour. Chem. Soc., **64**, ii, 46; Ztschr. angew. Chem., 1892, 600; Vjschr. Chem. Nahr., **7**, 208; Exp. Sta. Record, **4**, 85. J. W. F.

II.—CRUDE SALT.—ZINC OR COPPER METHOD.

62. EHRMANN, L., January, 1893.—*Determination of potassium by reduction of potassium-platinic chlorid* (*Dosage de la Potasse par Reduction du Chloroplatinate de Potasse*).

Reference is made to the leading methods for potash estimation, and especially to the Lindo-Gladding method, which has been found highly satisfactory, both as regards ease of execution and accuracy of results. It was desired, however, to devise a method which was more rapid than those in ordinary use, and which at the same time would avoid the excessive washing of the precipitate with alcohol and also the subsequent drying.

The author recommends proceeding as in the sodium formate process until the precipitate, with the accompanying impurities, has been brought upon the filter (and the excess of platinic chlorid, of course, washed out). The potassium-platinic chlorid is then dissolved out with hot water, and to the hot solution a small excess of zinc dust is added, acidity of the liquid during the reduction being avoided.

The reducing action of the zinc is quite rapid, and after the reduction is complete the liquid is decanted and the residue treated with very dilute acid to dissolve the excess of zinc.

The precipitated platinum, after washing, is ignited for an instant and then weighed. Freshly precipitated copper and cuprous oxid also reduce the double platinum salt very readily.

Bull. Assoc. Chim. France, **10**, 536. B. B. R.

II.—CRUDE SALT (CONTINUED).—MAGNESIUM METHOD.

63. VILLIERS, A., and BORG, F., June, 1893.—*The determination of potassium* (*Dosage du Potassium*).

The processes ordinarily employed for the estimation of potassium, by weighing the double platinum salt on a tared filter, or by reduction of the precipitate in a current of hydrogen and washing out the potassium chlorid, are either somewhat open to error or are tedious and time consuming.

The following process can be executed with ease and rapidity, and gives accurate results: The double chlorid of potassium and platinum, obtained in the usual manner, is thoroughly washed with a mixture of equal volumes of alcohol and ether. The precipitate is next dissolved in boiling water and to the solution thus obtained is added a little pure hydrochloric acid. Small particles of magnesium are now successively introduced until the liquid is completely decolorized. The precipitated platinum is very easily washed and does not adhere to the sides of the vessel. After being brought upon the filter it is washed, dried, ignited, and weighed. In calculating the results the factor 0.3939 is employed for determining the proportion of potassium, and the factor 0.4747 for the amount of K_2O.

This mode of reduction furnished a good substitute for sodium formate in the sodium-formate method, since, in this latter process, the platinum is very finely divided and adheres very strongly to the sides of the vessel in which precipitated. The method described has proved very successful in the estimation of potash in mixtures of the sulphate of potash with phosphate and sulphate of soda, and the chlorids of calcium, magnesium, iron, etc.

Compt., rend. **116**, 1524. B. B. R.

4. *Bitartrate method.*

64. BAYER, A., May, 1893.—*The determination of potash and soda by the bitartrate method* (*Ueber directe Bestimmungen von Kali und Natron mittels der Bitartrat Methode.*)

The usual method for potash determinations is somewhat long and tedious on account of the necessity for the removal by precipitation of certain acids and bases which are present. The author, in consequence, proposes a process for the determination by the bitartrate method which is applicable in the presence of sulphates, phosphates, chlorids, etc. About 15 grams of substance are dissolved in 500 cc. water; to 25 cc. of this in a beaker, about 0.3 gram of crystallized sodium carbonate is added and then 6 cc. of 80 per cent acetic acid and 2.4 grams crystallized tartaric acid. The solution is now heated, and after cooling 11 cc. absolute alcohol is added and the liquid is allowed to stand for two hours. The solution is filtered, 30 cc. of 25 per cent alcohol added to the contents of the beaker, and after mixing well the liquid is again filtered off; the precipitate is further washed with 50 per cent alcohol until 5 cc. of the filtrate show a scarcely appreciable trace of acidity. The precipitate, along with the filter paper, is titrated in a boiling solution with N/20 caustic soda, using phenolphthalein as indicator. To the figure thus obtained is added a correction for the proportion of potassium bitartrate soluble in the volume of alcoholic wash-water employed. The results quoted by the author for a number of analyses of potash salts show a close agreement with the platinum method. It is necessary to expel completely all ammonia salts before executing this process. The process for determining sodium depends upon the fact that in the presence of ammonium bitartrate in an alcoholic solution the amount of potassium left in solution is only one-third of that which dissolves under normal conditions. The alcoholic filtrate from such a solution, after evaporation, ignition of residue, resolution and removal of superfluous acids and bases as usual, yields a residue which, on ignition, is weighed as KCl+NaCl. From this is subtracted an amount of potassium chlorid equivalent to only one-third of the amount of potassium bitartrate which would dissolve in the alcoholic liquid under ordinary conditions.

Chem. Ztg. **17**, 686. B. B. R.

5. Perchlorate method.

65. Wense, W., 1892.—*Estimation of potassium as perchlorate.*

The author (Jour. Chem. Soc., **64**, ii, 46) has proved that adding a small quantity of perchloric acid to alcohol obviates its solvent action on potassium perchlorate, and affords a means for its estimation. If sulphates are present, the following modification is suggested: The solution is evaporated with perchloric acid to decompose the chlorids, and the residue is extracted with acid alcohol, and finally dissolved in boiling water. The solution is evaporated in a platinum dish, the residue calcined to convert the perchlorate into chlorid, then dissolved in water, and titrated with a standard solution of silver nitrate.

Ztschr. angew, Chem., 1892, 233; 234; Jour. Chem. Soc., **64**, ii, 190. J. W. F.

66. Caspari, R.—*Wense's method for the determination of potash* (*Ueber Darstellung der Ueberchlorsäure und Kalibestimmung mittels Ueberchlorsäure bei Gegenwart von schwer- und nichtflüchtigen Säuren*).

Caspari finds, after repeated trials, that Wense's method applied to potash salts is both accurate and convenient. He gives detailed instructions for the preparation of perchloric acid in considerable quantity and at a slight expense. Wense's method may be used for fertilizers containing phosphates of lime, iron, and the other bases usually encountered, as these salts are soluble in alcohol in the presence of excess of perchloric acid. In such cases the author proceeds as follows:

The hydrochloric acid solution of the fertilizer is evaporated until the free acid is removed, the residue is treated with 20 cc. of hot water, and perchloric acid added equivalent to at least one and one-half times the quantity of bases present. The solution is evaporated to a sirupy consistency, hot water added, and the evaporation continued with repeated stirring until the odor of hydrochloric acid is no longer perceptible and the white fumes of perchloric acid begin to come off. If much of the latter is removed it should be replaced. When cold the more or less sirupy residue is gently stirred into a homogeneous mass, 20 cc. of wash alcohol (97 volume per cent containing 0.2 per cent of its weight of perchloric acid) added, and the stirring continued, taking care not to rub up the crystals into a fine powder. When the supernatant liquid is clear it is decanted on an asbestos filter. The alcohol is removed from the residue by heating gently, 0.3 gram of perchloric acid and a little water are added and the residue washed on the filter with alcohol wash, and finally with a little pure alcohol. The entire wash liquid need measure but 35 to 70 cc. Mixtures of $CaCO_3$, Mn_3O_4, Fe_2O_3, Al_2O_3, Na_2HPO_412Aq, $MgCl_2$, H_3PO_4 yielded by this method a residue weighing but 0.0011 to 0.0016 gram, while on mixtures of KCl with H_3PO_4 or HNO_3 in different proportions the potash was practically all obtained as perchlorate.

When sulphuric acid is present the author proceeds as follows: Five grams of the substance are boiled with 150 to 180 cc. water and 20 cc. conc. hydrochloric acid, and during the boiling barium chlorid solution (prepared by dissolving 127 grams crys. barium chlorid in water, adding 125 cc. 35 per cent hydrochloric acid and making up to 1,000 cc.) is added, drop by drop, until in excess, the boiling being continued some minutes longer. When cool the solution is made up to 250 cc., filtered, and an aliquot portion treated as already described. Results, accurate.

Ztschr. angew. Chem., 1893, 43; Ber. chem. Ges., **26**, Ref. 335. A. L. W.

6. Spectroscopic method.

67. GOOCH, F. A., and HART, T. S.—*Detection and estimation of potassium spectroscopically.*

An effort is made to improve the method by the substitution of more powerful flames and coils of platinum wire instead of loops. The quantitative estimation is made by testing a standard solution of potassium in the apparatus and diluting the liquid under examination until its potassium line is of the same brightness as that given by the same quantity of the standard solution. The presence of sodium causes a much brighter potassium line, and introduces a difficulty. The results are not satisfactory, except for small amounts of potassium.

Amer. Jour. Sci., **42**, 448–459; Jour. Chem. Soc., **62**, 913, 914. J. W. F.

NITROGEN.

REAGENTS.

1. Standard acids, etc.

68. RICHMOND, H. DROOP—*Normal sulphuric acid, barium sulphate, and decinormal barium hydrate.*

Description of methods of preparing the standard solutions given, with the precautions to be observed in the determination of barium as $BaSO_4$.

Analyst, **17**, 167, 168. F. W. W.

2. Standardizing agents.

I.—POTASSIUM TARTRATE.

69. BORNTRÄGER, A., 1892.—*Use of potassium hydrogen tartrate in standardizing alkalis (Saures weinsaures Kalium als Ausgangspunkt für die Acidimetrie).*

The author recommends the above salt as useful in the preparation of standard alkaline solutions.

Ztschr. angew. Chem., 1892, 294; Jour. Chem. Soc., **64**, ii, 144; Ztschr. anal. Chem., **31**, 43. J. W. F.

II.—POTASSIUM DICHROMATE.

70. BREUTEL, E.—*Potassium dichromate as a standard for acidimetry (Kaliumbichromat als Urmaass für die Acidimetrie).*

The coarsely powdered substance is heated, with constant stirring, until it becomes brown and commences to fuse. After cooling in desiccator, weigh about 2 grams, dissolve in a little water, add some phenol-phthalein solution, and run in baryta water of one-fourth to one-fifth normal strength, until a change of color is effected. The appearance of the liquid is similar to that noted with silver solution and potassium chromate. At first the color is brown-red, but after sitting ten hours the supernatant liquid becomes red. From the result of the experiments the alkalimetric value of the baryta water is calculated, and by aid of this an acid can be readily standardized.

Ber. Oesterr. Ges., **16**, 142; Chem. Ztg. Rep., **16**, 329; Vjschr. Chem. Nahr., **1**, 492. B. B. R.

III.—Potassium tetroxalate.

71. Parsons, C. L.—*Comparison of methods for the standardization of acid and alkaline solutions.*

See Proceedings of this Association, 1892, p. 80. Jour. anal. Chem., **6**, 372–375; Jour. Chem. Soc., **64**, ii, 232. A. L. W.

72. Hinman, Bertrand C.—*Potassium tetroxalate as a standard in acidimetry and alkalimetry.*

A quantity of the salt prepared by combining proper proportions of potassium oxalate and oxalic acid and recrystallizing four times was found to contain the theoretical per cent of potash but an excess of acid, thus rendering it unfit for standardizing. Phenolphthalein is the best indicator, methyl orange being unsatisfactory, and coralline, cochineal, and Congo red worthless. Water can not be determined by heating. At temperatures above 100° C. the salt decomposes, although Bornträger (Ztschr. anal. Chem., **31**, 43) says it must be heated at 128° to insure perfect drying. When containing its full amount of water of crystallization it shows a tendency to effloresce.

School of Mines Quarterly, 1892, July; Jour. anal. Chem., **6**, 435. A. L. W.

IV.—Borax.

73. Rimbach, E.—*Employment of borax for standardizing acids.*

The author recommends the employment of borax, with methyl-orange as indicator, in place of sodium carbonate. Recrystallizing commercial borax two or three times, and drying in the air, sufficiently purifies the salt for average purposes. One gram of borax is equivalent to 5.2391 cc. of normal acid, or 1 liter of normal acid corresponds with 190.872 grams of crystallized borax.

Ber. chem. Ges., **26**, 171; Jour. Chem. Soc., **64**, ii, 233; Rev. internat. falsificat., **7**, 62. J. W. F.

V.—Copper sulphate.

74. Hart, Edward.—*Copper sulphate as a material for standardizing solutions.*

The method described by Hart and Croasdale (Jour. anal. Chem., **4**, 424; U. S. Dept. Agr. Bull., 31, 124, also 130) gives accurate results. T. W. Richards (Proc. Amer. Acad., **26**, 240) finds the ratio between Cu and H_2SO_4 to be 1:1.54197. Richards (Proc. Amer. Acad., **26**, 245) and Hampe (Ztschr. anal. Chem., 13, 352) have shown that copper sulphate may be easily purified by adding to the solution a trace of potassium hydrate to precipitate bismuth and iron, and ammonium bromid to throw down silver. Richards (Chem. News, 43, 22) finds that with methyl-orange a minute excess of acid can be detected. Pure copper sulphate may be prepared by dissolving electrolytic copper in hot concentrated sulphuric acid. A black residue of copper sulphid remains undissolved. The addition of alkali sulphate to increase conductivity is unnecessary.

Jour. anal. Chem., **6**, 421. A. L. W.

75. Drossbach, G. P.—*Electrolytic estimation of copper.*

The author recommends electrolyzing an ammoniacal solution instead of the usual acid solution of copper, the end of the process being more easily recognized.

Chem. Ztg., **16**, 819; Jour. Chem. Soc., **64**, ii, 93. J. W. F.

VI.—Ammonium sulphate method.

76. Weinig, M., 1892.—*Gravimetric estimation of sulphuric acid.*

An accurately measured quantity of the acid is mixed with a slight excess of ammonia, evaporated to dryness, dried for half an hour at 115° to 120°, cooled in a desiccator, and weighed as ammonium sulphate. The results are said to be more accurate than those obtained by precipitation with barium chlorid.

Ztschr. angew. Chem., 1892, 204, 205; Jour. Chem. Soc., **64**, ii, 145. J. W. F

3. *Iodid acidimetric method.*

77. BAUMANN, A.—*Acidimetry without normal alkali* (*Acidimetrie ohne Normallauge*).

The reaction which takes place when a free acid is brought in contact with a mixture of potassium iodate and iodid can be utilized in the determination of the amount of free acid gas-volumetrically. About 1 to 2 grams of the iodate and 10 grams of the iodid are brought into the generating chamber of the azotometer employed, dissolved in water, and the acid liquid is then run in, it being necessary that the latter be largely diluted. The glass cylinder attached to the apparatus is charged with hydrogen peroxid and caustic potash; the stopper is now replaced, the mixed liquids run into the generator, and the oxygen evolved is collected in the usual way; from this volume, properly corrected, the amount of free acid is calculated. Acids in only very weak solutions can be estimated in this way.

Ztschr. anal. Chem., **31,** 450; Chem. News, **66,** 254. B. B. R.

4. *Indicators.*

78. ALLEN, A. H.—*Neutrality.*

An exhaustive discussion of the various indicators used in titrations.

Analyst, **17,** 186–192, 215–219. F. W. W.

79. BREED, MARY BIDWELL, February, 1893.—*On phenolphthalein and methyl-orange as indicators.*

Author gave results indicating neutral reaction of phenolphthalein to acid carbonates, to which methyl-orange is acid and should therefore be used.

Jour. Frank. Inst., **135,** 312, 313. W. F

80. DENIGES, G.—*Preservation of solution of m-phenylenediamine.*

This reagent may be preserved, if dissolved in 50 parts of ammonium hydrate, by shaking with animal char and kept in contact therewith in a well-stoppered bottle.

Jour. pharm. chim. [5], **25,** 591; Bull. Soc. Chim. [3], **7,** 717, 718. W. F.

81. LÜTTKE, J.—*Sensitive litmus indicator.*

One hundred grams of litmus are extracted with warm water, and the extract, concentrated to 200 cc., is mixed with 20 cc. of 25 per cent hydrochloric acid and dialyzed until the acid is removed.

Ztschr. anal. Chem., **31,** 692; Jour. Chem. Soc., **64,** ii, 294. J. W. F.

82. THOMSON, R. F., May, 1893.—*Phenolphthalein as an indicator.*

The author refers to the difficulty of arriving at a satisfactory end reaction in titrating solutions of certain acids, as sulphurous, boric, arsenious, etc., with phenolphthalein as an indicator. The degree of dilution in a number of cases has been found to affect the results of the titrations; if, for instance, a solution of borax, which has been apparently neutralized by sulphuric acid, is diluted with water, the pink color will return and an appreciable quantity of acid will be required to discharge the color.

A reverse case is noted with regard to strong ammonium sulphate, additional quantities of sodium hydrate being required to produce the pink color after dilution of the solution.

The theory is advanced by the author that, in the cases mentioned a partial decomposition has taken place on dilution and that the solution is either acid or alkaline according as the acid or the base is the stronger.

It was found that, as in the case of soaps, the normal acidity of solutions of boric acid could be restored by adding a considerable proportion of glycerol (30 per cent) to the solution, the results thus secured corresponding closely with the theoretical figures and the end reaction being very distinct. Cane sugar and starch glucose in solution were also found to cause a very considerable increase in the amount of normal alkali used in titrating boric acid solutions.

Jour. Soc. Chem. Ind., 12, 432, 433; Ber. chem. Ges., **26,** R, 839, 840. B. B. R.

ANALYSES BY COMBUSTION.

Dumas's method.

83. O'SULLIVAN, J.—*Dumas's method of estimating nitrogen in organic substances.*

The author's experience proves that when a plug of asbestos is substituted for the copper gauze not much more nitric oxid is found in the resultant gas than when the copper is used. He recommends determining the amount of nitric oxid and subtracting it from the total volume.

Jour. Soc. Chem. Ind., **11** (1892), 327, 328; Jour. Chem. Soc., **64**, ii, 297; Ber. chem. Ges., **25**, Ref. 804. J. W. F.

84. BLAU, F.—*Method of determining nitrogen in organic substances.*

A modification of Dumas's method is proposed, the chief objections to the original process being the difficulty of exhaustion of air from the copper oxid, its inapplicability to liquids, and the necessity of refilling the tube for each analysis. The combustion tube is connected at one end with the delivery tube, and at the other end (which is also open) with a T tube. One limb of the T connects with a gas generator, the third opening being closed with a cork; by the employment of this connection, a new boat can be introduced without permitting the entrance of more than a trace of air. The tube is filled in the following manner, commencing with the forward end: Copper oxid, 8 cm.; copper, 12 cm.; copper oxid, 35 cm.; while the space occupied by the boat and the substance to be burned is 25 to 30 cm.

The copper oxid is granulated, and the copper is obtained from the oxid by reduction. Both the copper and the oxide are heated to redness in a current of carbon dioxid until the gas has been absorbed by caustic potash. The substance under examination is then heated strongly, and the combustion completed in a stream of oxygen, the last trace of nitrogen being swept out by means of carbon dioxid. The carbon dioxid is generated in an apparatus of special construction by allowing a very strong solution of potassium carbonate to flow into 50 per cent sulphuric acid. The potassium carbonate is preferred to the sodium compound on account of the small amount of air it dissolves.

The oxygen required in the process is generated by the action of hydrogen dioxid, in the presence of dilute sulphuric acid, upon potassium dichromate.

Monatsh. f. Chem., **13**, 277; Jour. Soc. Chem. Ind., **12**, 185; Ber. chem. Ges., **25**, Ref. 603. B. B. R.

Sodium formate method.

85. ARNOLD, C., and WEDEMEYER, K.—*Estimation of nitrogen in nitrates.*

In a combustion tube 45 cm. long and 10 to 12 mm. bore, place 5 cm. of a mixture of 1 part sodium formate with 9 parts of soda lime, then the substance with enough of a mixture of equal parts of sodium formate, soda lime, and crystallized sodium thiosulphate with 2 parts of dehydrated thiosulphate to occupy 25 to 28 cm.; then 10 cm. of the mixture of soda lime and formate. Keep the receiver cool, and heat until the last trace of water is distilled over. Use fluoresceïn or lacmoid as an indicator, so that the hydrogen sulphid may have no influence. The method is not applicable to nitro compounds, nitrites, or nitrates of pyridin and chinolin.

Ztschr. anal. Chem., **31**, 389-392; Jour. Chem. Soc., **62**, 1517, 1518; Exp. Sta. Record, **4**, 583; Chem. Centralbl., 1892, ii, 672; Ber. chem. Ges., **26**, Ref. 154; Vjschr. Chem. Nahr., **7**, 355. J. W. F.

ANALYSES IN THE WET WAY.

A.—ORGANIC SUBSTANCES.

1. Kjeldahl method.

86. ARNOLD, C., and WEDEMEYER, K.—*Kjeldahl's nitrogen method* (*Beiträge zur Stickstoffbestimmung nach Kjeldahl*).

Since the introduction of Kjeldahl's method for nitrogen determination, many modifications have been proposed from different sources for the purpose of accelerating the destruction of organic matter during the digestion.

By combining the modifications of Gunning and of Arnold, the oxidation of the organic matter can be effected much more expeditiously than by any process previously employed. The employment of potassium sulphate, mercuric oxid and copper sulphate along with sulphuric acid not only permits the digestion to be completed in a much shorter time than by the usual methods, but also allows of the determination of nitrogen in many organic substances for which the other modifications can not be utilized. Among these substances are the azo, chinolin and pyridin compounds. The proportions of the substances employed in this method are: 0.5 gram of sample, 30 to 50 grams of H_2SO_4, 15 to 25 grams of potassium sulphate, and 1 gram each of mercuric oxid and copper sulphate. On account of the considerable foaming which takes place when large quantities of potassium sulphate are present, only a portion of this substance is employed at first, the remainder being added after ten or fifteen minutes.

It is claimed that by this method the time of digestion is shortened from one-half to one-third, the results of numerous experiments being quoted to substantiate this claim.

For the determination of nitrogen in the nitrates of both organic and inorganic bases, salicylic or benzoic acid is added to the sulphuric acid (3 grams per 40 cc. H_2SO_4). This is heated for a short time in the flask along with the sample, and then the remaining substances, with exception of K_2SO_4, are added, the latter being introduced after ten to fifteen minutes' boiling.

Ztschr. anal. Chem., **31**, 525–533; Jour. Chem. Soc., **64**, ii, 88; Chem. Centralbl., 1892, ii, 1045; Ber. chem. Ges., **26**, Ref. 158; Vjschr. Chem. Nahr., **7**, 493; Rev. internat. falsificat. **7**, 83, 84. B. B. R.

87. Böttcher, O.—*Estimation of nitrogen by Kjeldahl's method.*

The author prefers the use of mercury instead of copper sulphate in the analysis of highly nitrogenous organic matter, and states that potassium sulphid and zinc are not necessary before distilling, 1.5 grams of zinc dust being sufficient.

Landw. Vers.-Stat., **41**, 170–173; Jour. Chem. Soc., **64**, ii, 144; Chem. Centralbl., 1893, i, 130; Exp. Sta. Record, **4**, 583; Ber. chem. Ges., **26**, R, 511. J. W. F.

88. Petit, A., and Monfet, L.—*Quick determination of organic nitrogen* (*Schnelle Bestimmung des organischen Stickstoffs*).

The process proposed is essentially that of Kjeldahl with certain modifications and is especially adapted to the analysis of wine. The digestion process is conducted very much after the manner of Kjeldahl's, sulphuric acid and metallic mercury being employed. When cool, a little water is added to the contents of the flask and then caustic soda solution until the solution is still left slightly acid. The liquid is then made up to a definite volume and an aliquot part is treated with alkaline hypobromite solution in a graduated vessel; the nitrogen is collected in the usual way and after reading off its volume the results are corrected for temperature and pressure.

Jour. pharm. chim. (5), **27**, 297; Chem. Ztg. Rep., **17**, 78; Chem. Centralbl., 1893, i, 856; Jour. Chem. Soc., **64**, A, 343. B. B. R.

89. Lawes, E.—*Determination of nitrogen by Kjeldahl method.*

Finds that, contrary to previous reports, the nitrogen in glucosazon, phenylhydrazin, and antipyrin can not be determined by the Kjeldahl method.

Pharm. Ztg., **37**, 552, 553; Chem. Centralbl., 1892, ii, 628. E. W. A.

90. Edwards, V.—*Estimation of nitrogen.*

The use of an iron flask for distillation in the Kjeldahl method is recommended as rapid, convenient, and advantageous, but a small correction must be made for the nitrogen absorbed by the moist iron oxid and given off during the distillation. For nitrates the author employs the iron flask, and 10 grams of zinc grains, 5 grams of iron filings, 0.5 gram of the nitrate, 100 cc. of a 30 per cent solution of potash, and 50 cc. of water. After an hour 100 cc. more water is added and the ammonia distilled into standard acid. Blank determinations should, of course, be made with the reagents.

Chem. News, **65**, 241–265; Jour. Chem. Soc., **62**, 1125; Ber. chem. Ges., **25**, 650; Ztschr. angew. Chem., 1892, 600; Vjschr. Chem. Nahr., **7**, 205. J. W. F.

2. Gunning method.

91. Huguet, M.—*Determination of nitrogen (Die Bestimmung des gesammten Stickstoffs).*

Author proposes to let the nitrogenous liquid enter drop by drop into the hot mixture of sulphuric acid and potassium bisulphate (5 cc. and 10 grams, respectively) in a Kjeldahl digestion flask, the liquid to be admitted no faster than the color is destroyed. When all has been added heat until a clear straw tint is obtained, and add distilled water drop by drop to bring the whole contents into solution. Cool, transfer, and distill as usual in the Kjeldahl process. Use Poirrier's orange No. 3 as indicator. Substances as difficult of decomposition as quinin may be analyzed thus.

Jour. pharm. chim. [5], **26**, 54, 56; Bull. Soc. Chim. [3], **7**, 717; Chem. Ztg. Rep., **16**, 243. W. F.

3. Manganese-dioxid method.

92. Stock, W. F. K.—*A new and rapid method for the determination of nitrogen in organic bodies.*

Five-tenths to one gram of substance, according to amount of carbonaceous matter to be oxidized, is mixed in a conical flask or small beaker with 10 cc. H_2SO_4 (sp. gr. 1.84) and 5 grams MnO_2, which has been made to pass through a sieve with 36 holes to the inch. The flask is heated on an iron plate until the contents assume a dark-green color. The flask is then cooled, cold water added, and after having been transferred to an apparatus described in the paper, the contents are distilled with an excess of a sodium-hydrate solution (20 grams for 10 cc. H_2SO_4). Comparative results given, most of which agree closely. Attempts to use the method for the determination of N in ferrocyanid and some alkaloids failed.

Analyst, **17**, 109–113; Exp. Sta. Record, **4**, 86. F. W. W.

93. Stock, W. F. K.—*Supplementary notes on a new and rapid method for the determination of nitrogen in organic bodies.*

Analyses of pure NH_4Cl and of potassium ferrocyanid by the method are given; results very satisfactory. Time needed for the oxidation three to four minutes.

Analyst, **17**, 152, 153. F. W. W.

94. Stock, W. F. K.—*The Stock process for the determination of nitrogen.*

The distillation apparatus is modified and now consists of a Wurtz flask of not more than 150 cc. capacity, the tube from which runs into the condenser, and it is kept at boiling point by the ammoniacal steam from the distillation flask.

Analyst, **18**, 58; Ber. chem. Ges., **26**, Ref. 390. F. W. W.

95. Skertchley, W. P.—*Notes on Stock's nitrogen process.*

Analyses given by the new method and by the Kjeldahl method. Crystallized ammonium sulphate gave satisfactory results, and also wheat flour and commercial gelatin; coffee, cotton meal, linseed cake, crushed horns and hoofs, fish guano, dried blood, and manure all gave more or less unsatisfactory and always too low results. Time of oxidation from ten to fifteen minutes. Author concludes that the method needs a great deal more examination before its adoption can be recommended.

Analyst, **17**, 209–215.

96. Stock, W. F. K.—*The Stock method for the rapid determination of nitrogen.*

A reply to Mr. W. P. Skertchley (Analyst, **17**, 209–215; see above). Author claims that Mr. Skertchley did not follow instructions in working the method, the manganese dioxid used containing only 40.86 per cent MnO_2 and the substance having not been pulverized sufficiently.

Analyst, **18,** 58–60. F. W. W.

97. ZAY, C. E.—*The estimation of organic nitrogen by Stock's method.*

Staz. sper. agrar. Ital., **26,** 22–31; Chem. Centralbl., 894, i, 746.

B.—MIXED ORGANIC AND NITRATE SUBSTANCES.

1. Modified Gunning method.

98. WINTON, A. L.—*Modification of the Gunning-Kjeldahl method applicable in presence of nitrates.—On the Gunning-Kjeldahl method and a modification applicable in the presence of nitrates.*

The material, 0.5 to 1.0 gram, is digested with 30 cc. of Scovell's salicylic acid mixture (30 cc. of sulphuric acid and 2 grams salicylic acid) in a flask of 600 cc. capacity for two hours with frequent shaking. Two grams of zinc dust are then slowly added, with continual shaking, and the flask heated, at first gently, until after a few minutes' boiling dense fumes are no longer given off. Thus far the process is the same as in the Scovell-Kjeldahl method, except that the digestion is continued for two hours, which in some cases is necessary in order to secure complete solution of the nitrate. Ten to 12 grams of potassium sulphate are next added and the boiling continued for a little time after the solution is colorless, or, if iron is present, has a light straw color, which remains unchanged. On cooling, as the mixture begins to solidify, water is added, at first slowly with shaking, and the distillation with caustic soda is carried on in the usual manner. The average of the 25 determinations by the Scovell-Jodlbauer method is 4.64 per cent, by the proposed method 4.65. The greatest discrepancy in any instance is 0.10 per cent, the average discrepancy 0.05 per cent. In 10 cases the proposed method gave the lower result, in 13 cases the higher result.

Bull. 112, Conn. (State) Agl. Exp. Sta., 1892, June; Chem. News, **66,** 227; Ztschr. angew. Chem., 1893, 200; Ber. chem. Ges., **26,** Ref., 294; Exp. Sta. Record, **4,** 336; Jour. Chem. Soc., **64,** ii, 145. A. L. W.

2. Jodlbauer-Kjeldahl modification.

99. CHENEL, L.—*Estimation of nitrogen in nitrates and nitro-compounds by Kjeldahl's method.*

The author finds that if phosphorus iodid instead of zinc dust be employed to effect the reduction in Jodlbauer's modification, the nitrogen in nitronaphthalene may be accurately estimated.

Bull. Soc. Chim. [3], **7,** 321–327; Jour. Chem. Soc., **64,** ii, 87, 88; Exp. Sta. Record, **4,** 286. J. W. F.

3. Combined Ulsch and Kjeldahl processes.

100. SCHENKE, V., 1893.—*Determination of nitrogen in nitrates and in mixtures of nitrates with organic and inorganic nitrogenous materials (Stickstoffbestimmung in Nitraten, sowie eine neue einfache Modification zur Bestimmung des Gesammtstickstoffs in Gemischen von Nitraten mit organischen und anorganischen Stickstoffverbindungen).*

After a critical review of the numerous methods proposed for the determination of nitric nitrogen, the author selects the Ulsch method as being the best on account of its simplicity, rapidity, and accuracy. He finds it very essential that the proportions between substance and reagents be correct, and recommends the following: In the analysis of sodium nitrate 20 grams of substance are dissolved in 1 liter of water, and to 25 cc. of this solution in a distilling flask are added 4 grams of reduced iron and 12 cc. of sulphuric acid (sp. gr. 1.35). This mixture is gradually heated to boiling, where it is maintained a few minutes, and then after washing down the inside of the flask 50 cc. of water and 25 cc. of sodium hydrate (sp. gr. 1.3) are added. The ammonia is then distilled over and titrated as usual.

He also recommends the following combination of the Ulsch and Kjeldahl methods for the analysis of mixtures containing nitrates: From 1 to several grams of substance are placed in a digestion flask, mixed with water to a pasty consistency, from 1 to 4 grams of iron added, according to the content of nitrogen, and from 5 to 10 cc. of sulphuric acid (sp. gr. 1.35). The mixture is then digested carefully until the end of the reaction. A little copper oxid and about 15 cc. of sulphuric acid, containing 200 grams of phosphoric anhydrid per liter of concentrated acid, are then added and the mixture is again digested as in the Kjeldahl method until the solution becomes light green. The digestion occupies from three-quarters to an hour and a quarter, when much organic matter is present. The addition of a few grains of zinc will prevent bumping.

Chem. Ztg., **17,** 977–979; Exp. Sta. Record, **5,** 222; Jour. Soc. Chem. Ind., **12,** 1063; Ber. chem. Ges., **26,** Ref. 830. J. P. S.

C.—NITRATES.

1. Boyer method.

101. ARNOLD, C., and WEDEMEYER, K.—*Boyer's method of nitrogen determination (Ueber die Bestimmungsmethode des Stickstoffs nach E. Boyer).*

Boyer's process for determination of nitrogen in nitrates, as described in Compt. rend., **113,** 503, consists in the combustion of the substance with 50 parts of a mixture of 1 part of sulphur, 2 parts of calcium oxalate, and 6 parts of soda lime. Experiments were made by the authors with the nitrates of potassium, sodium, and silver, and the results secured neither agreed with the theoretical percentages nor with check determinations of nitrogen in the same substance. Numerous variations in the mixing proportions of the above substances yielded no better results, even with addition of sugar, sodium thiosulphate, etc., and the method is hence pronounced entirely useless for the purposes for which it was designed.

Ztschr. anal. Chem., **31,** 388; Jour. Chem. Soc., **62,** 1517; Ber. chem. Ges., **26,** Ref. 154; Chem. Centralbl., 1892, ii, 671. B. B. R.

2. Copper-iron method.

102. ULSCH, K.—*Method of estimating nitrogen in nitrates and nitrites.—Action of the copper-iron couple on nitrates and nitrites in sulphuric and hydrochloric acid solutions (Ueber die Wirkung des Eisen-Kupfer auf Nitrate und Nitrite in schwefelsaurer und chlorwasserstoffsaurer Lösung).*

The author has improved his method so that now the operation is carried out as follows: The liquid containing the nitrates and nitrites is mixed with 10 cc. of normal sulphuric acid and 10 cc. of a 10 per cent solution of crystallized copper sulphate, and is made up to 100 cc. Twenty cubic centimeters of this serves for each determination. The reaction flask is charged with 3 grams of iron powder, and the air is expelled by adding 10 cc. of 3 per cent sulphuric acid. The flask is then connected with the azotometer, 2 cc. more of the acid is run in, and then rinsed in by two additions (5 cc. each) of the mixture to be assayed. The flask is then plunged for two minutes into a water bath at 60°, and is then shaken vigorously for two minutes. The solution is then drawn off and the flask cooled, and all is ready for the estimation. Twenty cubic centimeters of the above mixture are run in. Nitrites reveal their presence by producing the black solution of nitric oxid. The flask is shaken in the cold until the black color just disappears. It is then warmed to 60° and shaken again. The deficit in the volume of hydrogen evolved is the sum of the deficits due to the nitrates and nitrites present. If it be wished to know the amount of each separately, the nitrite must be estimated by other means.

Ztschr. anal. Chem., **31,** 392–404; Ber. chem. Ges., **26,** 155; Jour. Chem. Soc., **62,** 1518; Vjschr. Chem. Nahr. **7,** 356. J. W. F.

103. Ulsch, K., 1891.—*Estimation of nitrogen in nitrates.*

The author recommends placing in the receiver a quantity of standard acid slightly insufficient to neutralize the ammonia evolved on distillation as giving more trustworthy results than the usual procedure.

Ztschr. angew. Chem., 1891, 719; Jour. Chem. Soc., **64**, ii, 42. J. W. F.

3. Zinc-iron methods.

104. Märcker, M.—*The direct determination of nitrogen in Chile saltpeter.—Kuehn's zinc-iron method commended.*

Landw. Vers.-Stat., **41**, 365; Chem. Centralbl., 1893, i, 501; Jour. Chem. Soc., **64**, ii, 237, 238.

105. Schmitt, T. F., February, 1892.—*The determination of nitrogen in nitrates (Bestimmung des Stickstoffs in Salpeter und Salpetergemischen).*

The method proposed is an improvement upon the original one described in the Chemiker-Zeitung (**14**, 1410), and which provided for the reduction of the nitrates by means of powdered zinc and iron in the presence of acetic acid. Into a globular flask of about 750 cc. capacity are brought 10 grams of glacial acetic acid and 8 to 10 grams of the mixed powdered metals; after shaking, 25 cc. of a solution of the nitrate (equivalent to 0.5 gram) is added. To avoid any mechanical loss, the flask is covered during the evolution of gas. This ceases in about ten minutes, and after dilution with 200 to 300 cc. water, 30 cc. of caustic soda solution of sp. gr. 1.25 is added and the distillation conducted as usual. The fineness of division of the iron powder employed influences appreciably the rate of reduction of the nitrates. The whole analysis is completed in forty to fifty minutes.

By employing this process in conjunction with the Kjeldahl method, the total nitrogen of guanos and nitrate mixtures can be determined. One gram of substance dissolved in water is employed, and to this are added 5 cc. glacial acetic acid and 2 to 3 grams of the metallic mixture.

After reduction in the usual manner and cooling the flask, 25 cc. concentrated H_2SO_4 is added gradually and with care, excessive frothing being avoided by the use of paraffin. The acetic acid is driven off by heating, and the digestion and distillation performed as in the method of Kjeldahl.

The process has been already successfully employed for two years.

Chem. Ztg., **17**, 173; Analyst, **18**, 144; Chem. Centralbl., 1893, i, 582; Jour. Chem. Soc., **64**, A, 434. B. B. R.

106. Robineau, F., and Rollin, G.—*The presence of ammonia in zinc dust (Ueber die Gegenwart von Ammoniak im Zinkstaub).*

Commercial zinc dust appears to invariably contain ammonia—a fact which must lead to errors where this substance is employed in the conversion of nitrates into ammonia compounds. By treating zinc dust with warm water a trace of ammonia can be readily detected in the liquid by means of Nessler's reagent. The thoroughly washed zinc dust will, moreover, yield ammonia when boiled with caustic, although the last of the washings with water may have shown no reaction with Nessler's solution. The ammonia can be removed from the zinc dust by washing the latter with 1 per cent boiling sulphuric acid until Nessler's test gives no reaction. This then no longer gives off ammonia on boiling with caustic alkali. If this powder is dried in ammonia-free air, it will, however, after two days, be found to contain ammonia. For analytical purposes zinc should be treated as described and used at once.

Monit. Scient. [4], **7**, 138; Chem. Ztg. Rep., **17**, 40; Analyst, **18**, 144, 145.

B. B. R.

107. Wedemeyer, K., 1893.—*Note on the estimation of nitric nitrogen by the Schmitt method (Beitrag zur Stickstoffbestimmung in Nitraten nach der Schmitt'schen Methode).*

The method has been tested in samples containing nitrates and has been found satisfactory.

Arch. Pharm., **231,** 372–376; Ber. chem. Ges., **26,** Ref. 728; Chem. Centralbl., 1893, **2,** 288; Jour. Chem. Soc., **64,** A, 551. J. P. S.

4. *Iron method.*

108. BÖTTCHER, O.—*Determination of nitric nitrogen.*

Ten grams of saltpeter are dissolved in 500 cc. H_2O; 25 cc. of the solution are transferred to a 400 cc. Kjeldahl distilling flask with about 120 cc. water; about 5 grams zinc dust (previously washed and dried), 5 grams iron (powdered "ferrum limatum pulv.," not "ferrum reductum"), and 80 cc. soda solution (sp. gr. 1.3) are then added, the flask is connected with a distillation apparatus and 20 cc. standard H_2SO_4 added in the receiving flask. After standing for one or two hours with repeated shaking, about 100 cc. are distilled over, and the distillate titrated as usual. Results with this and Jodlbauer method agreed closely, both for pure saltpeter, Chile saltpeter, and mixtures of various fertilizers, superphosphate, bone meal, meat scraps, fish guano, etc. Same method described in Landw. Vers.-Stat. (**41,** 370) with apparatus devised by G. Kuehn.

Landw. Vers.-Stat., **41,** 165–169; **42,** 129; Jour. Chem. Soc. **64,** ii, 145; Chem. Centralbl., 1893, i, 129; Exp. Sta. Record, **4,** 583; Ber. chem. Ges. **26,** R, 511.

F. W. W.

5. *Alloy method.*

109. DEVARDA, A., December, 1892.—*A direct determination of nitrogen in nitrates* (*Ueber die directe Bestimmung den Stickstoffs im Salpeter*).

The author presents the following as a quicker method than those of Böttcher and Stutzer. Ten grams of saltpeter are dissolved in 1 liter of water, and 50 cc. transferred to a 600 to 800 cc. Erlenmeyer flask with about 60 cc. water, 5 cc. alcohol, and 50 cc. potash solution (sp. gr. 1.3), 2 to 2½ grams of a pulverized alloy of Al 45 parts, Cu 50 parts, and Zn 5 parts (made by T. Svpek, 62 Stumpergasse, Vienna) are then added, and the flask at once connected with a distillation apparatus; it is then heated slowly and after one-half hour distilled over. The distillation will take twenty minutes. The arrangement of the distillation apparatus adopted described in the paper and also precautions to be observed.

Landw. Vers.-Stat., **42,** 130–134; Chem. Ztg., **16,** 1952; Ber. chem. Ges., **26,** Ref. 337; Analyst, **18,** 92, 93; Jour. Chem. Soc., **64,** A, 343. F. W W.

6. *Iodometric method.*

110. GOOCH, F. A., and GRUENER, H. W.—*Iodometric estimation of nitrates.*

The authors recommend distilling the nitrate (about 0.2 gram) with 20 cc. of a saturated solution of crystallized manganous chlorid in strong hydrochloric acid, in a current of carbonic anhydrid. The products of the distillation are passed into a solution of potassium iodid, and the liberated iodin is afterward titrated with sodium thiosulphate. Three molecules of iodin correspond to two molecules of hydrogen nitrate.

Amer. Jour. Sci. [3], **44,** 117–123; Jour. Chem. Soc., **64,** ii, 42; Jour. anal. Chem., **6,** 40; Ztschr. angew. Chem., 1893, 199. J. W. F.

111. GRUENER, H. W.—*Iodometric estimation of nitrates.*

Two processes are described: (1) Reduction by means of phosphoric acid and potassium iodid; (2) reduction by means of antimonous chlorid. The test analyses are reported to be fairly satisfactory.

Amer. Jour. Sci., **46,** 42–50; Jour. Chem. Soc., **64,** A, 550. J. W. F.

7. *Modified Schlösing method..*

112. GLASER.—*Gasometric estimation of nitric acid.*

In those methods which depend on the reduction of the nitric acid by hydrochloric acid and ferrous chlorid, and measurement of the nitric oxid, there is always a liability to error from the presence of dissolved oxygen in the liquid over which the gas is collected, whereby part of the nitric oxid is converted into nitrous acid. This can be prevented by collecting the gas over a 1 per cent solution of potassium iodid, when the well-known reaction between nitrous and hydriodic acids, resulting in the liberation of iodin, and nitric oxid occurs. If carbonates are present, the gas must be treated with soda.

Ztschr. anal. Chem., **31,** 285–288; Jour. Chem. Soc., **62,** 1375; Ber. chem. Ges., **25,** Ref. 873. J. W. F.

113. DE KONINCK, L.—*A new way of carrying on the Schlösing method for the determination of nitric nitrogen.*

Ztschr. anal. Chem., **33,** 200–205; Chem. Ztg., **18,** Repert., 128; Chem. Centralbl., 1894, i, 1011; Ber. chem. Ges., **27,** Ref. 344.

8. Cinchonamin method.

114. GAMMARELLI, P.—*Determination of nitric acid as cinchonamin nitrate.*

Arnaud's method gives low results because of the solubility of cinchonamin nitrate in water.

Atti. R. Acc. d. Lincei 1892, II sem., 290–294; Gazz. Chim. Ital., **22,** ii, 635; Ber. chem. Ges., **26,** Ref. 103; Chem. Centralbl, 1893, i, 130. A. L. W.

9. Amalgam method.

115. MONNIER, D., and AURIOL, H., 1894.—*Volumetric estimation of nitric acid.*

The reduction of the nitrate takes place in an acid solution by means of sodium amalgam and acidified water. The apparatus used is too complex for description here.

Arch. sc. Phys., Genève, **31,** 352–358; Chem. Centralbl., 1894, i, 1095, 1096. J. P. S.

10. General nitrate methods.

116. ALBERTI and HEMPEL.—*Determination of nitrogen in Chile saltpeter (Die quantitative Bestimmung des Stickstoffs im Natronsalpeter).*

The author indorses the methods of Lunge, Schlösing-Grandeau-Wagner, and Ulsch. High results are obtained when sodium nitrate is obtained by difference after determining water, matter insoluble in water, sodium sulphate, and sodium chlorid.

Ztschr. angew. Chem., 1891, 398–400: 1892, 101. Ber. chem. Ges., **25,** Ref. 593. Jour. Chem. Soc., **64,** ii, 42. C. L. W.

117. STOKLASA, JULIUS.—*Comparative tests of methods for the determination of nitrogen in nitrates (Beitrag zur Bestimmung des Stickstoffs in Kali-Natron-Salpeter und in Salpetersäure).*

The methods investigated are as follows:

(1) Indirect method for analysis of Chile saltpeter. Moisture, matters insoluble in water, chlorin and sulphuric acid were determined directly, and the sodium nitrate and nitrogen calculated from these data.

(2) Wagner's modification of the Schlösing-Grandeau method.

(3) Lunge's nitrometer.

(4) Stutzer's method.

(5) Devarda's method. (Chem. Ztg., **16,** 1952.) In applying this method the author proceeds as follows: 50 cc. of a solution of the nitrate (equivalent to 5 grams) are placed in a copper Erlenmeyer flask and diluted with 200 cc. of water, 40 cc. potassium hydrate (sp. gr. 1.277), 5 cc. alcohol, and 2.5 grams finely divided Devarda's alloy are then added, and the flask connected with the distilling apparatus. After heating gently for twenty minutes, the reduction is complete, and the ammonia thus formed is distilled over by boiling twenty minutes longer and titrated. Devarda's alloy is prepared by adding 5 parts of zinc to a molten mixture of 50 parts of copper and 45 parts of aluminum.

The following results were obtained on pure potassium nitrate and commercial nitrate of soda.

	Indirect method.	Schlösing-Grandeau.	Lunge's nitrometer.	Stutzer.	Devarda.
Potassium nitrate c. p.:					
Average of 4 determinations		13.77	13.85	13.90	13.91
Commercial nitrate of soda:					
Sample A	15.82	15.60	15.59	15.61	15.62
Sample B	15.75	15.52	15.54	15.60	15.58
Sample C	15.69	15.50	15.45	15.51	15.59

From these analyses the author concludes that all the methods investigated, excepting the indirect method, give accurate results. He regards Lunge's nitrometer best suited for rapid work, only thirty to forty minutes being required for a determination. Results are also given on samples of nitric acid illustrating the accuracy of Lunge's, Stutzer's, and Devarda's methods.

Ztschr. angew. Chem., 1893, 161–163; Chem. Centralbl., 1893, i, 857; Jour. Chem. Soc., **64,** A, 389; Jour. Soc. Chem. Ind., **12,** 711. A. L. W.

118. SCHRAM A.—*Analysis of Chile saltpeter.*

Oesterr.-ungar. Ztschr. Zuckerind., 1892, 684, 685; Chem. Centralbl., 1892, ii, 628.

D.—NITRITES.

119. GROSSMANN, J.—*Estimation of nitrites.*

The author bases his method upon the fact that if a nitrite be boiled with dilute sulphuric acid, only two-thirds of the base is taken up by the acid. The acid is standardized with a pure nitrite as follows: 3 grams of pure sodium nitrite are dissolved in 200 cc. of hot water, and after adding 40 cc. of normal sulphuric acid, boiled until all nitric oxid has been expelled. The excess of acid is then titrated with normal soda. If the nitrite to be tested contains free alkali, this must be separately estimated and allowed for.

Chem. Ztg., **16,** 818, 819; Jour. Chem. Soc., **64,** ii, 87. J. W. F

120. REITMAR, O., and STUTZER, A.—*Estimation of nitrous acid in sodium nitrate.*

The authors conclude that the permanganate method gives erroneous results, and urge the adoption of Lunge's process for accurate determinations. (Compare Jour. Chem. Soc., **62,** 1029.)

Ztschr. angew. Chem., 1891, 666, 667; Jour. Chem. Soc., **64,** ii, 43. J. W. F.

E.—MISCELLANEOUS.

121. AUFSCHLÄGER, H.—*A reaction for nitrogenous organic substances* (*Ueber eine Reaction stickstoffhaltiger organischer Substanzen*).

A new reaction for nitrogenous organic substances is reported by the author, it being especially adapted to testing for urea, uric acid and derivatives, albuminous substances, etc. When nitrogenous substances of this character are mixed with zinc dust and heated in a combustion tube, among other products formed is zinc cyanid, which can be examined in the usual way.

Monatsh. f. Chem., **13,** 268; Ztschr. anal. Chem., **32,** 95. B. B. R.

122. HENRY, E.—*The determination of nitrogen with the aid of the nitrometer* (*Note sur le dosage de l'azote nitrique à l'aide du nitromètre*).

The article requires the accompanying illustrations.

Bull. Soc. Chim., **11** [**3**], 24–27; Ber. chem. Ges., **27,** R, 207; Chem. Centralbl., 1894, i, 393, 394. W. D. B.

123. Ilosva, L. I. de N.—*The estimation of ammonia, nitrous and nitric acids by the colorimeter.*

Bull. Soc. Chim., **11** [3], 216–226; Chem. Centralbl., 1894, i, 746; Ber. chem. Ges., **27**, R, 345.

124. Koninck, L., March, 1893.—*Testing for ammonia with Nessler's reagent* (*Zum Nachweis des Ammoniaks mit Nessler'schem Reagens*).

The observation has been very recently made that Nessler's reagent, so sensitive under ordinary conditions, gives in the presence of ammonia in alcoholic solutions neither a yellowish brown precipitate nor a coloration. The presence of alcohol, however, does not interfere with the reaction with mercuric chlorid (Bohlig's test).

Ztschr. anal. Chem., **32**, 188; Chem. News, **69**, 220, 221; Chem. Centralbl., 1894, i, 1096; Ber. chem. Ges., **26**, R, 506. B. B. R.

125. Neumann, B., June, 1893.—*Detection of ammonia with Nessler's reagent* (*Zum Nachweis des Ammoniaks mit Nessler'schem Reagens*).

Issue is taken with the statement of Koninck (Ztschr. anal. Chem., **32**, 188), in which it is claimed that Nessler's reagent, in the presence of ammonia, in alcoholic solutions gives no coloration nor precipitate. The results of a number of tests are reported, in which a trace of ammonia in alcoholic solutions of varying concentration, gave a very marked reaction in every case, even with pure absolute alcohol. The erroneous results of Koninck's tests are ascribed to an error in the preparation of the reagent or to having allowed the solution to stand for too long a time.

Chem. Ztg., **17**, 880. B. B. R.

1. Detection of thiocyanates.

126. Jumeau, P. L.—*Estimation of ammonium thiocyanate in nitrogenous manures.*

In presence of chlorids, after the sulphates are removed, the thiocyanates may be estimated by oxidation with potassium permanganate and hydrochloric acid and weighed as barium sulphate. In absence of chlorids they may be titrated with silver nitrate, using ferric sulphate as indicator. In the presence of both cyanids and chlorids, they may be titrated against potassium permanganate in the presence of sulphuric acid.

Jour. Pharm., **27** (5), 190–193; Jour. Chem. Soc., **64**, A, 347. J. W. F.

2. Detection of leather.

127. Lindsey, J. B., April, 1894.—*Leather refuse; its value in agriculture. Detection of leather in steamed and acidulated fertilizers.*

In a paper discussing the subject as a whole, certain matters of analytical interest are included. The author has found Dabney's (N. C. Exp. Sta. Bull., 3, 1883) solution of phosphate of iron in phosphoric acid efficient to detect leather in fertilizers. It was further found that pepsin-pancreas digestion, according to Stutzer's method, on coarse leather, sole leather finely ground, and the same heated for some time with superheated steam gave percentage of digestibility varying from 52 to 97, while pepsin digestion alone resulted in the solution of only 13 to 42 per cent. After these several leathers were acted upon by sulphuric acid the coefficient of pepsin digestion rose to 54 to 71 per cent, indicating much higher availability.

Agric. Science, **8**, 98–108. F.

SOIL ANALYSIS.

General.

128. Kühn, J.—*Examination of soils* (*Bodenuntersuchung*).

Report of the committee on examination of soils of the Association of German Experiment Stations. The report recommends the adoption of a 2-mm. sieve for sampling of "fine earth" for chemical analysis. Reference is made to the original paper for details in manipulation of mechanical soil analysis.

Landw. Vers.-Stat., **42**, 153–160; Chem. Centralbl., 1893, i, 229. F. W. W.

129. O'Brine, David.—*Methods of soil analysis.*

The methods of Hilgard and Peter are described in detail.

Jour. anal. Chem., **6**, 541. A. L. W.

130. Gundlach, G.—*Methods of analysis of moorland soils.*

Dry substance: The fresh sample is air-dried at ordinary temperature, and then pulverized and carefully mixed. Ten grams of the air-dry sample are then dried in a tared weighing tube in a drying oven for five or six hours at 100° to 110° and again weighed.

Ash: 100 grams are first heated in a platinum dish (holding 50 cc.) at low red heat for a good while, and the incineration is then finished by frequent stirring with a platinum spatula. The crude ash is weighed, and 1 gram of the same taken for CO_2 determination by Wagner–Knop's azotometer. The total crude ash is boiled with dilute HCl (Mn present, if Cl-gas is generated; in which case the boiling is continued until the smell of Cl disappears). The mass is then transferred to a filter; the residue after washing, drying, and weighing represents sand and clay, uncharred carbon being present only in trace. The solution and wash water are made up to 200 cc. and serve for further determinations

Potash and soda determinations: 100 cc. of the solution are taken; the sum of chlorids first determined, and then the potash, by precipitation with $PtCl_4$.

P_2O_5 determination: 40 cc. of the solution are taken, and the phosphoric acid determined by the molybdenum method.

SiO_2: Another 40 cc. of the solution taken for this determination and proceeded in the usual manner.

In the filtrate from the latter determination Fe_2O_3, Al_2O_3, and P_2O_5 are precipitated by ammonia at boiling temperature; the precipitate ignited and weighed, and the phosphoric acid, already determined, deducted. In the filtrate from this determination the lime is precipitated at boiling heat by ammonium oxalate, filtered after standing for twelve hours, and ignited over blast lamp until constant weight.

Magnesia is determined in the filtrate from this determination as $Mg_2P_2O_7$; a slight coloration of the latter shows the presence of a trace of Mn.

Nitrogen is determined by the modified Kjeldahl method; 1 gram of the air-dry material is heated with about 20 cc. conc. H_2SO_4, with addition of some kernels of CuO, some $PtCl_4$, and a trace of paraffin (free from N) until a clear solution is obtained, and the analysis is then finished in the ordinary manner, one-fifth HCl being added in the receiving flask and $Ba(OH)_2$ solution (50.25 cc. = 30 cc. of the HCl sol.) used for titrating back the excess of HCl.

For the determination of total H_2SO_4 in the ash, 10 grams of the air-dry sample is moistened with a 3 per cent Na_2CO_3 solution in a platinum dish by means of a pestle; it is then dried on a water bath and charred over an alcohol flame; the ash treated with water and filtered; the residue and filter dried and incinerated over alcohol flame. The filtrate and ash are then united, and after an excess of HCl is added and the solution boiled and filtered, the sulphuric acid is precipitated with $BaCl_2$ and determined in the ordinary way.

Jour. f. Landw , **40**, 232–236. F. W. W.

131. Hilgard, E. W.—*Chemical and physical investigation of soils.*

In this paper the importance of observation of the soil in place, the correct taking of the sample and the preliminary tests, is urged. The "soil" comprises all to the lower limit of the humus color line. It is first examined macroscopically and microscopically for undecomposed minerals. The first examination is a study of their surface condition, in the coarse residue left after washing ten grams in a water current of definite velocity. Then the "hand test" is given by crushing small lumps between the thumb and forefinger in the dry condition first, then wet and observing the change in color; then kneading to test the contents of plastic clay.

The maximum and minimum water capacity is determined in a cylindrical brass vessel with perforated bottom, of 25 to 50 cc. capacity; the dry soil sifted in, weighed and placed in a shallow dish with a layer of water for an hour, when increase in weight gives amount of water held. Then the wet soil is covered with air-dried soil, then with such as has been saturated with moisture until the weight ceases to decrease. This gives its minimum capacity.

Hygroscopic moisture is determined by exposing a layer of the soil 1 mm. thick for twelve hours to an atmosphere saturated with moisture (in a box) at about 15° C. Transfer to a U tube and quickly weigh; dry in a bath at 200° C. and loss gives the moisture. At a temperature of 100° soils will continue to lose water for a week. From the drying tube the amounts are weighed out for analysis. That for general analysis is digested with chlorhydric acid, sp. gr. 1.115; that for phosphoric acid in nitric acid, in porcelain beakers over a steam bath for five days and evaporated to complete dryness over sand bath. The analysis is then made by the usual silicate method as fully outlined in the annual report of the California Station for 1892. For humus the usual Grandeau method is used. For nitrogen determination potash or soda solution is used instead of the ammonia in that method for the leaching out of the humus; the filtrate is evaporated to dryness after being neutralized and the nitrogen determined by the Kjeldahl method.

A discussion of results follows the brief outline of method of analysis. Comparison with cultural experience seems to show that the amount of available plant food in virgin soils of essentially similar origin is sensibly proportional to the respective totals of such ingredients found by analysis; provided that we take into consideration both the proportions of these ingredients among themselves and the physical nature of the soil.

Jour. Amer. Chem. Soc., **16**, January. L. AND J.

132. HILGARD, E. W.—*Solvents for soil analysis*

This paper embraces a discussion of the relative merits of organic and mineral acids in soil digestion, and in porcelain and glass beakers. The author maintains that in order to obtain results available for discussion in comparison with results of culture, the maximum solvent power of plant acids should be made of use; and that in this respect oxalic acid may be considered as equal to chlorhydric or nitric.

Agric. Science, **7**, 427–432. L. AND J.

133. HILGARD, E. W.—*Digestion of soils for analysis.*

In this paper the author discusses the behavior of chlorhydric acid on soils under evaporation in the process of five days' digestion, as a test of the supposition that in the watch-glass-covered beakers the acid is concentrated and its solvent power changed. The changes in strength of the acid are by the tests shown to be insignificant, and that evaporation during soil digestion is effective in maintaining or restoring a definite strength, independently of the amount of matters dissolved, as against closed-flask digestion according to Kedzie. The importance of correct sampling and description of soil samples is then urged and examples given.

Agric. Science, **8**, 1–4. L. AND J.

134. DYER, BERNARD, February 15, 1894.—*The analytical determination of probable available "mineral" plant food in soils.*

The methods pursued and the results obtained in the investigation are given in detail. The root acidity in the case of different orders of plants varied from 0·34 per cent (calculated as citric acid) in *Solanaceæ* (potato) to 3.4 per cent in *Rosaceæ*, and averaged 0.91 per cent for the twenty orders. The results were thought to indicate that the 1 per cent citric acid solution proposed by Tollens and Stutzer for the determination of reverted phosphoric acid in manures would probably meet the required conditions, and it was decided to test it on soils of known history and recorded fertility. The method used in determining citric acid, soluble potash, and phosphoric acid was as follows: A weight of air-dried soil, corresponding to 200 grams of completely dried soil, was placed in a bottle with 2 liters of distilled water, in which was dissolved 20 grams of pure citric acid. The soil was allowed to remain in contact with the 1 per cent citric acid solution for seven days, being, except on one day, shaken up a great many times each day—i. e., whenever the soil had settled well down. The temperature ranged between 10° and 19°, and averaged about 15° C.

After the treatment as above the solutions were filtered. Five hundred cubic centimeters, that is, the solution from 50 grams of soil, was used for each determination. In each case the solution was evaporated to dryness in a platinum basin and gently incinerated at a low temperature. The residue was dissolved in pure chlorhydric acid, evaporated to dryness, redissolved, and filtered. In the filtrate the phosphoric acid, or potash, as the case might be, was determined by the Hehner and Tatlock methods, and as the actual precipitate weighed represented 50 grams of soil, multiplication of any experimental error in converting into percentages was minimized.

The general conclusion was reached that the use of the citric-acid solution gives valuable indications of the available fertility of soils such as are not obtained in ordinary soil analysis, and the results suggest that when a soil is found to contain as little as about 0.01 per cent of phosphoric-acid soluble in a 1 per cent solution of citric acid it would be justifiable to assume that it stands in immediate need of phosphatic manure. The potash supply of soils appears to be more complicated than that of phosphoric acid and needs further investigation. The results in the present case indicate that the limit marking the nonnecessity of special potash applications probably lies below 0.005 per cent of citric-acid soluble potash.

Jour. Chem. Soc., 1894, **65**, 115–117; Chem. News, **69**, 13, 14; Exp. Sta. Record, **5**, 1013–1015; Agric. Science, **8**, 75–86. L. AND J.

MECHANICAL ANALYSIS.

135. HILGARD, E. W., November, 1892.—*Mechanical soil analysis.*

The author discusses at length the advantages of boiling in the preparation of the sample and of his churn elutriator for the prevention of flocculation and for the separation of silts of different hydraulic value.

Agric. Science, **6**, 502–509. W. F.

136. HILGARD, E. W., June, 1893.—*Sedimentation versus hydraulic elutriation.*

A reply to T. B. Osborne, advocating the latter method, and dealing with details of construction of the churn elutriator.

Agric. Science, **7**, 278–284. W. F.

137. OSBORNE, THOMAS B., April, 1893.—*Mechanical soil analysis.*

A reply to Dr. Hilgard as regards the relative merits of pestling and boiling in the preparation of samples, and as regards the beaker *versus* the elutriation method.

Agric. Science, **7**, 187–192. W. F.

138. HOLLEMAN, A. F.—*Determination of sand in soils.*

Schoene's elutriating apparatus as modified by Ad. Mayer was used, arranging the same so that 1 liter of water flowed out of the opening in ten minutes by a pressure of 5 cm., while the elutriation itself was conducted by a pressure of 2 cm.

Landw. Vers.-Stat., **41**, 38. F. W. W.

MECHANICO-CHEMICAL ANALYSIS.

139. TSCHAPLOWITZ, F., September, 1892.—*The determination of sand and clay in soils.* (*Ueber die Bestimmung von Thon und Sand im Boden*).

The estimation of clay in soils is sometimes accomplished by washing out the fine particles with water, decomposing the clay with sulphuric acid, and calculating the amount equivalent to the alumina found. By another process, the clay is estimated by simple mechanical washing of the soil, though by this process too high results are obtained, since bodies other than clay, if of sufficient fineness, are washed out and weighed as clay: the figures obtained by this method are then in direct contrast to those secured by the sulphuric acid process, which yields too low results.

The method recommended as well adapted to soil analysis consists in first boiling some of the sample with water until the lumps disappear; hydrochloric acid of 10 per cent strength is next added to dissolve carbonates, more water and acid being added immediately thereafter. The whole is then boiled and allowed to stand for a day. After the liquid has been poured off, the residue is twice washed by decantation with warm water. The washed residue is then heated with 3 per cent ammonia to dissolve humus and afterward washed as before. The sediments from the acid and alkaline washings are collected, weighed, and reckoned as clay. The washed soil is now brought upon a sieve of 0.1 mm. mesh in a stream of water, and the coarse particles are ignited and weighed. The liquid which passes through the sieve is allowed to settle and is then drawn off. The purified soil is now placed in a cylinder and washed several times, the siphoned washings being allowed to settle or are evaporated to dryness, the residue being reckoned as clay. The coarse sediment› together with the particles separated by the sieve, is considered as sand.

Ztschr. anal. Chem., **31**, 487; Jour. Chem. Soc., **64**, ii, 97, 98; Ber. chem. Ges., **26**, Ref., 156.

CHEMICAL ANALYSIS—SPECIAL CONSTITUENTS.

A.—SULPHURIC ACID.

1. Barium Gravimetric method.

140. BROWNING, PHILIP E., 1893.—*The influence of free nitric acid and aqua regia on the precipitation of barium as sulphate.*

The author finds that the presence of an excess of nitric acid, or aqua regia, amounting to 10 per cent by volume of the liquid treated, is not harmful, but actually beneficial in estimating barium as sulphate. Under these conditions the precipitate falls coarsely crystalline, and is more easily filtered.

Amer. Jour. Sci., 1893, 399–404; Jour. Chem. Soc., **64**, A, 552. J. W. F.

141. RIPPER, M.—*Gravimetric estimation of sulphuric acid (Zur Gewichtsanalyse der Schwefelsaüre).*

The author recommends the treatment of the barium precipitate with bromin water, in order to oxidize any sulphid which may have been reduced by the carbon of the paper, to sulphate, and the subsequent washing of the precipitate with dilute hydrochloric acid in order to remove other substances which may have been carried down with the barium sulphate.

Ztschr. anorg. Chem., **2**, 36–44; Jour. Chem. Soc., **64**, ii, 239; Ber. chem. Ges., **26**, Ref. 161. J. W. F.

142. PHINNEY, J. I.—*Treatment of barium sulphate in analysis.*

The author recommends the collection of the precipitate on a Gooch's filter, so as to avoid reduction.

Amer. Jour. Sci., **45**, 468–472; Jour. Chem. Soc., **64**, A, 552. J. W. F.

2. Volumetric method.—Phosphoric acid.

143. BOURIEZ, A.—*Volumetric estimation of sulphates.*

The sulphate is boiled with a measured quantity of a standard solution of barium hydrogen phosphate, the barium being in excess. After filtering the barium sulphate, an excess of sodium carbonate is added to the filtrate, precipitating the barium as tri-barium phosphate. This precipitate is washed until neutral to phenolphthalein. The precipitate is dissolved in dilute hydrochloric acid, the barium precipitated by sodium sulphate, and the phosphoric acid estimated in the filtrate by Joly's method, which depends on the fact that methyl-orange reacts with phosphoric acid as a monobasic, while phenolphthalein reacts with it as a bibasic acid. The solution is then titrated with standard sodium hydroxid, using methyl-orange until the reaction occurs, then phenolphthalein is added, and titrated until the rose color appears. From the phosphoric acid found, the sulphuric acid may be calculated.

Rev. Internat. Sci. Fals. Aliment., **5**, 118; Chem. Centr., 1892, i, 570, 571; Jour. chem. Soc., **62**, 1377. J. W. F.

3. Volumetric method.—Chromic acid.

144. STOLLE, E , 1892.—*Volumetric estimation of sulphuric acid in sulphates* (*Massanalytische Bestimmung der Schwefelsaüre in verschiedenen Salzen*).

A weighed quantity of the substance is dissolved in water, and a hydrochloric acid solution of barium chromate is added until the sulphuric acid is precipitated. After adding a slight excess of ammonia, the whole is filtered and made up to 500 cc., and the chromic acid estimated in an aliquot by means of ferrous sulphate, the chromic acid serving as a measure of the amount of sulphuric acid.

Ztschr. angew. Chem., 1892, 234, 235; Jour. Chem. Soc., **64**, ii, 188, 189; Ber. chem. Ges. **25**, 596. J. W. F.

145. V. ASBOTH, A.—*Volumetric estimation of sulphuric acid in sulphates.*

The author has found that Stolle's process (Jour. Chem. Soc., **64**, ii, 188) does not give good results unless the barium chromate solution is freshly prepared, owing to the barium chlorid formed in the solution.

Chem. Ztg., **16**, 922; Jour. Chem. Soc., **64**, ii, 240. J. W. F.

B.—SULPHUR IN HUMUS SOIL.

146. BERTHELOT and ANDRÉ, 1892.—*Notes on the determination of sulphur in humus soil.* (*Quelques observations nouvelles sur le dosage du soufre dans la terre végétale, etc*).

Observe that the organic compounds of some soils hold their sulphur in such stable combinations that chlorin gas, acting either hot or cold, on the matter in the presence of potash, does not entirely oxidize it. These sulphur compounds are, however, almost entirely soluble in dilute, boiling potash solution; such a solution does not entirely decompose calcium sulphate.

Bull. Soc. Chim. [3] **7**, 435–437. W. F.

147. CARNOT, A.—*Estimation of phosphorus in soils.*

Ten grams of sifted soil, dried at 100°, is charred if much organic matter is present, moistened with water, and then with nitric acid until effervescence ceases, and then digested for two hours with 10 cc. of nitric acid at about 100°, replacing the acid evaporated, and stirring frequently. Wash on a filter with hot water, evaporate to 50 cc.; add 5 cc. of concentrated nitric acid and 0.5 gram of chromic acid crystals. Cover, and boil for half an hour. Add 5 grams of ammonium nitrate, and 50 cc. of ammonium molybdate solution, and keep at 100° for about an hour. Filter, and dissolve the precipitate in 30 cc. of ammonium hydrate diluted 1:1 with water. Neutralize with nitric acid, keeping the temperature below 40°; when the yellow precipitate ceases to dissolve on agitating the liquid, add a mixture of 3 cc. of pure nitric acid, 3 cc. of water, and 5 cc. of molybdate solution. After the precipitate has settled for two hours, it is filtered through a weighed filter, washed with 1 per cent nitric acid, and finally with a little pure water. Dry at 100° and weigh. The weight of the precipitate multiplied by 0.0373 gives the phosphoric acid in the 10 grams of soil.

Bull. Soc. Chim., **9** (3), 343–346; Jour. Chem. Soc., **64**, A, 489; Chem. Centralbl. 1893, ii, 147. J. W. F.

C.—CITRATE-SOLUBLE PHOSPHORIC ACID.

148. KUNZE, L.—*Determination of citrate-soluble phosphoric acid in soils* (*Untersuchungen der verschiedenen Bodenarten auf citratlösliche Phosphorsäure*).

Thirty grams of air-dried soil are placed in an Erlenmeyer flask with 300 cc. of 1 per cent citric acid solution and digested in the cold for twenty-four hours, the flask being loosely corked to allow escape of CO_2. Two hundred and fifty cubic centimeters of this solution are measured out and evaporated to dryness, a thick, sirupy mass being in most instances obtained. To this residue, 20 cc. strong H_2SO_4 and 5 cc. HNO_3 are added, and the dish and contents are heated over a Bunsen burner, observing due care during the foaming which takes place. The dry residue is treated with water, brought into a 250 cc. flask, and the solution made up to 250 cc. To 100 cc. filtrate, made weakly alkaline with ammonia, add a slight excess of HCl; cool and add 50 cc. citrate solution and 25 cc. magnesia mixture.

Ztschr. Ver. Rübenzucker, **42**, 862. B. B. R.

D.—IRON.

149. SACHSSE, R., and BECKER, A.—*Behavior of ferric oxid in soils and rocks.*

The authors estimate the free ferric oxid in soils by reducing with a stream of hydrogen in a platinum boat after the usual method, and measure the hydrogen evolved by its solution in sulphuric acid. Detailed process for mixtures of ferrous and ferric salts is given.

Landw. Vers.-Stat., **41**, 453–466; Jour. Chem. Soc., **64**, ii, 243, 244. J. W. F.

E.—CALCIUM.

150.—HOLLEMAN, A. F.—*Determination of lime in soils.*

Lime soluble in water.—Twenty grams of the air-dry soil are digested for forty-eight hours at ordinary temperature with distilled water, the volume being made up to 1 liter and contents frequently shaken. Five hundred grams of the filtrate are evaporated with a few drops of glacial acetic acid, the residue dissolved in water and a little acetic acid, and a measured quantity of a tenth-normal ammonium oxalate solution added. The solution is then made up to 100 cc., and after filtration the excess of oxalic acid in 50 cc. is determined by one-tenth permanganate solution.

Lime soluble in carbonic acid water.—Twenty grams of the air-dry sample are made up to 1 liter with distilled water, saturated with pure CO_2 gas, filtered after twenty-four hours, and the filtrate treated as above.

Landw. Vers.-Stat., **41**, 38. F. W. W.

151.—REVERDIN, F., and DE LA HARPE, C.—*Estimation of assimilable lime in soils.*

The method depends upon the fact that oxalic acid and its salts, like plant roots, attack only the surface of particles of calcium carbonate with which they come into contact. Determine total lime in soil as usual. Treat 20 grams of soil with 200 cc. of solution containing equal molecules of sodium carbonate and oxalate, in known quantity, and digest on water bath for some hours. Then surface of lime minerals is changed, but not of magnesia minerals. Filter, wash residue, acidulate combined filtrates with HCl. Filter off organic matter usually precipitated, add sodium acetate, precipitate residual oxalic acid by calcium chlorid, and in the precipitate determine oxalic acid by titration with permanganate and sulphuric acid. By difference, that taken up by the soil, and hence the corresponding assimilable lime, can be computed.

Chem. Ztg., **13**, 726; Ztschr. anal. Chem., **20**, 737; Bied. Centralbl., **23**, 410; Agric. Science, **8**, 197, 198. F.

F.—KAOLIN.

152. SACHSSE, R., and BECKER, A.—*Estimation of kaolin in arable soil.*

The soil is previously dried at 100° C. and extracted with dilute hydrochloric acid, and the dissolved sesquioxids and silica soluble in potash determined. These determinations are again made in second samples which have been gently ignited, and the *minus* of residue insoluble in acid and potash, or the *plus* of silica and sesquioxids which was obtained in the second experiment as against the first, calculated as kaolin. The composition of the mud is given as well as of other muds analyzed by the authors. (Compare Landw. Vers.-Stat., **38**, 411.)

Landw. Vers.-Stat., **40**, 245–255; Jour. Chem. Soc., **62**, 1026, 1027. J. W. F.

G.—SILICA

153. ARCHBUTT, L.—*Estimation of silica in clay.*

One gram of the finely powdered clay is fused with 5 grams of pure mixed alkali carbonates. The mass is extracted with about 200 cc. of water, the solution acidified with excess of hydrochloric acid, and evaporated to dryness on the steam bath in a porcelain dish, reducing the residue to small grains. It is then heated in an air oven at 150° for one hour, dissolved in hydrochloric acid and water, filtered, and the silica washed. The filtrate is evaporated with 20 cc. of sulphuric acid until fumes are evolved, the residue is dissolved in hydrochloric acid and water, and the additional silica collected and weighed. The two residues are ignited to constant weight, the silica volatilized by hydrofluoric acid and sulphuric acid, the residue ignited, and the difference is pure silica.

Jour. Soc. Chem. Ind., **11**, 215-217; Jour. Chem. Soc., **64**, ii, 300. J. W. F.

H.—NITROGEN.

154. LIEBSCHER.—*Determination of nitrogen in soils.*

Twenty to twenty-five grams of the samples of soils are weighed into flasks of 300 cc. capacity. Forty cubic centimeters H_2SO_4 containing 1 per cent phenol are then added, and also 1 to 2 drops of mercury and 1 gram zinc dust. The contents are then gradually heated until boiling point. The oxidation is finished within five or six hours, when 10 cc. H_2SO_4 are added. The ammonia formed is distilled in copper flasks under addition of NaOH and K_2S solutions, sufficient for neutralization of H_2SO_4 and Hg, respectively; 0.2 to 0.3 grams of zinc dust is added to prevent bumping.

Jour. f. Landw., **40**, 149. F. W. W.

155. MULLER, J. A.—*Determination of nitrogen in soils by Kjeldahl's method.*

Results by this method are only a trifle lower than by the soda-lime process if a little benzoic acid is added to the mixture of soil and fuming sulphuric acid. Fifteen grams of the dry, finely divided soil, 30 cc. of fuming sulphuric acid, and about 0.3 grams of benzoic acid are introduced into a flask with the neck so bent that when the flask is inclined the condensed vapor does not flow back. Heat twelve to fourteen hours, gradually raising the temperature until about 1.5 cc. of acid distil over.

Ann. chim. phys., (6) **22**, 393–401; Jour. Chem. Soc., **64**, ii, 43. J. W. F.

I.—HUMUS.

156. SNYDER, H., 1894.—*Notes on the Grandeau method for the determination of humus in soils.*

The author points out three serious objections to the Grandeau method as now used: (1) The difficulty in obtaining a complete extraction of the humus materials with ammonia, as ordinarily carried on, especially in very fine and clayey soils; (2)

the slowness of the filtration; and (3) the constant presence of ammonia fumes in the laboratory preventing other lines of work.

He offers the following modifications: After treatment with the dilute acid, the soil is transferred to either a glass-stoppered bottle, or a glass-stoppered Erlenmeyer flask of 100 cc. capacity, using 50 or 60 cc. of the dilute ammonia solution. The contents are shaken at frequent intervals and then allowed to settle. The dark-colored solution is then decanted into a filter, a fresh 50 cc. of the ammonia added to the flask, and the same treatment repeated. Three or four such treatments are usually necessary before the filtrate becomes clear. The contents of the flask are then brought on the filter, and require but little further washing in the usual way before the operation is completed. While the second, third, and fourth treatments with ammonia are being carried on the first portion of the filtrate can be evaporated on the water bath, thus saving considerable time. By making several determinations at one time much time can be saved. The results obtained are higher than by the usual way, which is to be expected, as the extraction is more complete.

In examining the results two points are to be noted: (1) There are many other organic compounds of equal agricultural value that are not included in the ammonia extract; (2) along with the humus soluble materials are variable amounts of phosphoric acid. The difficulty also presents itself that some of the phosphoric acid of the soil may be lost in the hydrochloric acid solution that is used to remove the lime.

He also finds that there is quite a close relation between the humus as determined by the modified method and the total nitrogen. The average of twenty-four analyses shows that there are about twelve parts of humus to one part of nitrogen.

Jour. Am. Chim. Soc., **16**, 210–213; Chem. Centralbl., 1894, **i**, 841; Exp. Sta. Record, **5**, 932. J. P. S.

157. BERTHELOT and ANDRÉ, 1894.—*Organic constituents of soils.*

Ann. chim. phys., **1** (7), 273–283; Chem. Centralbl., 1894, **i**, 433. J. P. S.

158. DE CHALMOT, G.—*Pentosans in soils.*

Amer. Chem. Jour., **16**, 229; Ber. chem. Ges., **27**, R, 422.

ASH ANALYSIS.

159. WEDEMEYER, K.—*The employment of calcium plumbate in ash analysis* (*Ueber die Verwendung des Calciumplumbats zu Aschenanalysen*).

The author reports as the result of practical experiments that the formation of calcium plumbate by heating calcium carbonate and lead oxid together does not take place so readily as has been stated. In experiments in ash determination the results are satisfactory so far as the quick combustion of the organic substances is concerned but there remains the objection that the regeneration of the calcium plumbate requires a very long time. By very greatly increasing the temperature the process can be accelerated, but in case chlorids are contained in the ash, there is danger of loss by decomposition or volatilization.

Arch. Pharm., **230**, 263; Ztschr. anal. Chem., **32**, 319. B. B. R.

160. BROWNING, PHILIP E., 1892.—*A method for the quantitative separation of barium from strontium by the action of amyl alcohol on the bromids.*

The author applies the above method for the separation of barium from strontium with fair results.

Amer. Jour. Sci., 1892, 459–462. J. W. F.

161. BROWNING, PHILIP E., 1892.—*A note on the method for the quantitative separation of strontium from calcium by the action of amyl alcohol on the nitrates.*

The author suggests the use of much smaller amounts of alcohol, thus requiring a smaller correction for solubility.

Amer. Jour. Sci., 1892, 462, 463 J. W. F.

162. Krüger, M.—*Estimation of small quantities of calcium.*

Hempel's method of titrating the oxalate against potassium permanganate is shown to be applicable to extremely small quantities of calcium.

Ztschr. physiol. Chem., **16**, 445–452; Jour. Chem. Soc., **62**, 914; Bull. Soc. Chim. [3], **8**, 1031. J. W. E.

163. Gooch, F. A., and Phinney. J. I., 1892.—*The quantitative determination of rubidium by the spectroscope.*

The authors apply the method of Gooch and Hart (Amer. Jour. Sci., XLIII, 448) to rubidium, and find that when only a few milligrams of rubidium are to be estimated the method is valuable and gives fair results.

Amer. Jour. Sci., 1892, 392–400. J. W. F.

164. Bayer, A.—*Direct estimation of potassium and sodium by means of tartaric acid.*

Sodium carbonate, sufficient to unite with any acids present, is added, then some acetic acid, and tartaric acid in excess. The liquid is warmed, mixed with one-third its volume of alcohol, and allowed to settle for two hours. The solution is filtered, the precipitate of potassium hydrogen tartrate washed slightly with 25 per cent alcohol, and then thoroughly with 50 per cent alcohol, dissolved in boiling water, and treated with decinormal soda, using phenolphthalein as indicator.

Sodium may be estimated in presence of potassium by boiling with ammonium hydrogen tartrate and a little acetic acid. When cold, the liquid is mixed with one-third of its volume of absolute alcohol, and later made up to 250 cc. with 25 per cent alcohol. An aliquot is filtered, evaporated with a little nitric acid, and gently ignited and extracted with water. The sodium is then weighed as chlorid.

Chem. Ztg., **17**, 686, 687; Jour. Chem. Soc., **64**, A, 598. J. W. F.

FOODS.

Vegetable Origin, but Including Honey.

A.—Carbohydrates.

1. General.

165. Stone, W. E., April, 1893.—*A review of recent investigations upon carbohydrates.*

The author presents summaries of recent discoveries concerning stachyose, fucose, agavose, lupeose, carbohydrate synthesis, the pentoses, dextrose, levulose, galactose, mannit and mannose, dulcit, rhamnose, sucrose, lactose, isomaltose, raffinose, perseit and starch; the phenomena of multirotation, calorimetric values, and analytical relations, the more important of which are noted elsewhere.

Agric. Science, **7**, 177–186. W. F.

166. Tollens, B.—*Experiments on carbohydrates.*

Landw. Vers.-Stat., **39**, 401–3; Ber. chem. Ges., **26**, R, 497.

167. Schulze, E., 1894.—*The quantitative determination of carbohydrates (Zur quantativen Bestimmung der Kohlenhydrate).*

Chem. Ztg., **18**, 527, 528; Chem. Centralbl., 1894, i, 930, 931.

168. Fischer, E., and Jennings, W. L.—*A resorcin test for carbohydrates.*

The authors state that resorcin and pyrogallol unite with glucose and arabinose, and other sugars, forming glucosidal compounds, and that these compounds give sharp color reactions with certain metallic bases in alkaline solution. Thus the reaction may be made a general test for carbohydrates. To 2 cc. of a dilute aqueous solution of the substance to be treated, add 0.2 gram of resorcin and saturate in the cold with HCl gas. If abundance of carbohydrates is probably present, let stand one hour, if only traces, twelve hours, at ordinary temperature. Dilute with water, add caustic soda in excess, and Fehling's in proportion to the carbohydrates present.

The reddish-violet color is very characteristic, but with great dilution disappears after some time. Insoluble substances are first rubbed fine, then extracted with water, and the extract tested. Test is not so good as Molisch's *a*-naphthol test, but valuable in confirmation.

Ber. chem. Ges., **27,** 1355–1362; Agric. Science, **8,** 199. F.

2. Sugar methods.—General.

169. FRESENIUS, W., and DOBRINER, P.—*Sugar determinations* (*Ueber Zuckerbestimmung*).

A résumé of the principal changes and improvements in the methods of sugar analysis during the past three or four years is presented in a condensed form, and the relative advantages of the more important processes are discussed at some length

Ztschr. anal. Chem., 706. B. B. R.

170. DONATH, E., 1893.—*Reaction of certain sugars with borax solutions* (*Notiz über die Reaction einiger Zuckerarten gegen Boraxlösung*).

Chem. Ztg., **17,** 1826.

171. LAVES, E., 1893.—*The quantitative and qualitative determination of sugars with phenylhydrazin.*

Arch. Pharm., **231,** 366–372; Chem. Centralbl., 1893, ii, 298.

172. *Methods used in enforcing the German sugar tariff* (*Zum Zuckersteuergesetz*).

The methods are described in detail. Formulæ and tables are given for calculation of results.

Ztschr. angew. Chem., 1892, 404. A. L. W.

3. Polarimetry.

I.—GENERAL.

173. FRIC, J., October 15, 1892.—*The manipulation of polarization apparatus* (*Ueber die Art der Behandlung der Polarisationsapparate mit Rücksicht auf ihre mechanische Einrichtung*).

A detailed description is given of the chief forms of polariscopic apparatus employed in sugar work, together with full illustrations by means of numerous plates and with directions for proper testing and adjustment. The quartz plate, accurately tested by employment of a polariscope with circular scale, is considered the most reliable standard for adjustment of instruments, and the use of four plates of values of about 25°, 50°, 75°, and 90° to 100° is recommended.

Böhm. Ztschr. Zuckerind., **17,** 7. B. B. R.

174. BÉCHAMP, A.—*The unchangeability of cane sugar in water solution at ordinary temperatures and the causes of the apparently spontaneous inversion under the influence of light.*

Bull. Soc. Chim., **9** [3], 21–27; Ber. chem. Ges., **26,** Ref. 542.

II.—SPECIFIC ROTATORY POWERS OF SUGARS.

175. KANONNIKOFF, J.—*Specific rotatory powers of sugars.*

Observations with a Landolt instrument indicate that when in a mixture of sugars the latter have the same sign of rotation, the rotatory power of the mixture is an exact mean of that of the component substances. When, however, the signs are opposite, the rotatory power of a mixture of the sugars is a multiple of the mean of the rotatory powers of the components. These multiples vary with the substances employed and with their number in the mixture.

Jour. Russ. Chem. Soc., **6,** 367–375; Bull. Soc. Chim. [3], **8,** 689, 690.

W. F.

176. O'SULLIVAN, 1892.—*Optical activity of levulose.*

The optical activity of levulose, as determined by calculation from the constants for invert sugar and dextrose, is announced as $[\alpha_j = -106^\circ$ or $(\alpha)_{l\,d} = -93.8$; these numbers agree fairly well with those of Ost $[(\alpha)_d = -93.01]$ and Tollens $[(\alpha)_d = -92.5]$
Chem. News, **65,** 165; Agric. Science, **7,** 181. W. F. S.

177. SCHULZE, C., and TOLLENS, B.—*Multirotation.*

Authors find that presence of 0.1 per cent of ammonia in the aqueous solution of the sugar entirely hinders multirotation of dextrose, galactose, arabinose, xylose, levulose, lactose, maltose, rhamnose, and half-rotating milk sugar. Stronger ammonia may diminish rotation, but only in unimportant degree. Such solutions color and decompose on standing.
Ann. Chem., **271,** 49–54; Landw. Vers.-Stat., **40,** 367–389; Centralbl. Agrik. Chem., **22,** 270; Exp. Sta. Record, **4,** 386; Ztschr. Ver. Rübenzucker-Ind., **42,** 747. W. F.

178. TOLLENS, B.—*Cause of birotation of dextrose* (*Ueber die Ursache der Birotation des Traubenzuckers*).

Ber. chem. Ges., **26,** 1799.

III.—MULTIROTATION.

179. SCHNELLE and TOLLENS, 1891.—*Multirotation of rhamnose and saccharin.* (*Ueber die Multirotation der Rhamnose und der Saccharine*).

The results of a series of experiments with regard to the polarization of rhamnose and saccharin under different conditions are given, and comparisons are made with results reported by other authors. The results with rhamnose, showing a gradual decrease in rotation with an increase in temperature, correspond closely with those of Rayman and Kruis, while the figures for saccharin differ quite materially from those obtained by other investigators. The specific rotary power of saccharin, which was found to be, at first, $(\alpha)\frac{20}{D} = 94.2^\circ$, after a period of eleven days had decreased to 88.7°. An increase in temperature also caused a decrease in the specific rotary power.
Ztschr. Ver. Rübenzucker-Ind., **42,** 744. B. B. R.

IV.—INFLUENCE OF PRECIPITANTS UPON LEAD SUBACETATE.

180. SAILLAND, C., October, 1892.—*The influence of lead subacetate upon polarization of cane juices* (*Controle chimique en sucrerie de canne*).

In the analysis of cane juices quite considerable differences in the polarization are noted on comparing the results secured by employment of normal lead acetate with those obtained by the use of lead subacetate as a clarifying agent. With the employment of the latter the results are invariably higher, owing to the formation of insoluble lead levulosate, which results in the increase of the dextrorotation of the solution. The author gives results of experiments illustrating these differences, and also figures showing the large discrepancies which can be occasioned thereby in sugarhouse control.

[NOTE BY ABSTRACTOR.—Gill and others called attention long since to the formation of the lead compound with levulose, which the author refers to.]
Bull. Assoc. Chim. France, **10,** 354. B. B. R.

181. COURTONNE, H., December, 1892.—*Subacetate of lead* (*Dēfinitions diverses du sous-acetate de plomb. Préparation nouvelle du sous-acétate de plomb*).

The author refers to the different processes recommended for the preparation of lead subacetate solutions by various authorities and to the differences in composition of the solutions thus obtained. A series of tables is given showing the proportions of subacetate of lead for solutions of different densities and also the amounts of neutral acetate, lead oxid, and acetic acid; in addition there are tables showing the densities of neutral acetate solutions, with percentages of acetic acid and also of lead oxid corresponding to those given in the subacetate tables. The author proposes the employment for sugar work of an easily prepared basic acetate solution which has been practically tested for a long period of time and has been found to give satisfactory results.

In the preparation of this solution the neutral acetate is dissolved in water and ammonia is added in such a proportion as will yield, by combining with a portion of the acetic acid, a basic lead acetate of the proper strength. The proportions of the substances employed are:

	Grams.
Neutral crystallized lead acetate	350
Distilled water	825
Ammonia (22°)	55

The ammonium acetate formed is without influence upon the results.

The author gives, in conclusion, a table showing the densities of solutions of this character, corresponding to varying proportions of subacetate.

Bull. Assoc. Chim. France, **10**, 457. B. B. R.

V.—Influence of precipitants upon alkaline nitrates.

182. Gravier, E., October, 1892.—*Influence of alkaline nitrates upon the polarization of sugar solutions* (*Influence des nitrates alcalins sur la polarisation des liquides sucrès.*)

The author gives the result of a number of tests with regard to the influence of potassium and sodium nitrates upon the polarization of sugar solutions. The nitrates were added to the solutions in the proportions of from 12.5 to 50 per cent of the sugar present, the polariscopic tests being made both upon aqueous and alcoholic solutions, and both with and without addition of lead subacetate.

In aqueous solutions, the presence of the nitrates exerted a practically inappreciable influence upon the polariscopic tests, both in presence and absence of lead subacetate. The influence was also imperceptible in alcoholic solutions, in absence of lead subacetate, but with addition of the subacetate solution and with 50 per cent of the nitrates a considerably lower polarization was noted. The author thinks it advisable, in consequence, to employ alcohol as little as possible as a solvent in sugar work.

Bull. Assoc. Chim. France, **10**, 351. B. B. R.

VI.—Effect of temperature.

183. Gravier, E.—*Influence of the temperature upon the polarization of sugar solutions.*

The author refers to the experiments of other investigators, notably Wartze, with reference to the influence of the temperature upon the polarization of sugar solutions, and urges as an objection to the conclusions of Wartze, that due correction for expansion and contraction has not been made. A series of experiments were conducted by the author in which the gaugings of the solutions were made at both 16° and 32° C., and each individual solution was also examined polariscopically at both of the above temperatures.

Instead of a variation of 0.1° of the polariscopic scale for each 2° C., as given by Wartze, it is claimed that the decrease should be only 0.1° for each 4° increase in temperature.

Bull. Assoc. Chim. France, **11**, 2. B. B. R.

184. Josse, A.—*Influence of temperature upon the rotation of cane sugar.*

Referring to the results reported by Gravier with reference to the influence of temperature upon the polarization of sugar solutions, the author reviews the results of Berthelot, Dubrunfaut, and others and also gives results recently secured by himself. He concludes that, for a difference in temperature of as much as 25° C., the variation in rotary power of a normal sugar solution is so small as to be practically inappreciable.

Bull. Assoc. Chim. France, **11**, 260. B. B. R.

4. Glucoses.

I.—GRAVIMETRIC ESTIMATION.

185. NIHOUL, E., April, 1893.—*The gravimetric estimation of reducing sugars* (*Ueber die gewichtsanalytischen Methoden zur Bestimmung reducirender Zucker*).

The method of Allihn, in which the precipitated copper suboxid is brought upon a weighed asbestos filter, dried, and reduced in a current of hydrogen, is a much more tedious and complicated process than a simple ignition of the precipitate on filter paper. The mode of procedure recommended by Holdefleiss provides for the ignition of the precipitate after oxidation with nitric acid and weighing as cupric oxid. Soxhlet has urged the objection against this process that a partly volatile basic copper nitrate is formed and also that a small quantity of copper remained absorbed in the filter paper even after repeated washing. The experiments made by the author to test the validity of the first objection showed that with careful and gradual heating no appreciable loss occurred, while any error arising from the second source of objection could be corrected by the employment of double filters. The filters used were of the same size and weight, and by placing one within the other it was assumed that the same amount of copper solution would be absorbed by each. The ash of the outer filter is subtracted from the weight secured by the ignition of the inner filter and contents after treatment with nitric acid. As the copper suboxid sometimes passes through the first filter, it may be found advantageous to employ two sets of double filters, igniting the first two along with the precipitate, and the second pair of filters, after being burned to ash, furnish a weight which is to be subtracted as before.

A simpler process is to employ dried filters of the same weight, the weight of the obtained cuprous oxid being ascertained by difference in weight of the outer filter and the inner filter and contents. This process yields exact results, since no oxidation of cuprous oxid has been found to take place, even after heating for two days at 75° to 100° C.

The author found, however, that a small proportion of organic matter is retained along with the precipitate, the amount so retained being equal to about 0.3 to 0.4 per cent of the weight of the precipitate. By making a correction for this, satisfactory results can be secured.

Chem. Ztg. **17**, 500; Chem. Centralbl., 1893, i, 96; Jour. Chem. Soc., **64**, A, 601.

B. B. R.

186. GRÜNHUT, L., 1894.—*The gravimetric methods for determining reducing sugars with alkaline copper solutions* (*Ueber die gewichtsanalytischen Methoden zur Bestimmung reducirender Zucker durch alkalische Kupferlösungen*).

Chem. Ztg., **16**, 447, 448; Chem. Centralbl., 1894, i, 878.

187. ROSS, B. B., February, 1893.—*Gravimetric estimation of invert sugar.*

The process described is a modification of the method of Formanek and is essentially that described in the Provisional methods for sugar analysis for 1892–'93. The electrolytic separation of the copper is effected in the beaker in which the precipitation is made, the asbestos filter and cuprous oxid being placed in the beaker without any attempt having been made to dissolve the latter. The anode employed is a flat platinum spiral which rests on the bottom of the beaker immediately underneath the asbestos, while a platinum cylinder receives the deposit of copper. The electrolysis is effected in a 4 per cent nitric acid solution, the cuprous oxid coming into solution continuously during the operation.

Jour. anal. Chem., **7**, 83.

188. Ehrmann, L., January, 1893.—*The gravimetric estimation of invert sugar* (*Dosage gravimétrique du sucre interverti*).

The gravimetric estimation of invert sugar can be readily accomplished by means of the reduction of the double chlorid of potassium or sodium and platinum by the cuprous oxid precipitated from Fehling's solution. The precipitated oxid, after being brought upon a filter and well washed, is treated with a few drops of a concentrated solution of sodium platinic or potassium platinic chlorid, and the reduction takes place at once on the filter. The proportion of invert sugar is calculated from the weight of the ignited platinum, and it is claimed that the results are more exact than by weighing the reduced copper, by reason of the considerably greater weight of platinum secured.

Bull. Assoc. Chim. France, **10**, 536. B. B. R.

189. Strieger.—*Determination of invert sugar by Soldaini's reagent* (*Dosage du sucre interverti avec le réactif de Soldaïni*).

The author makes the following modifications: Instead of weighing out 297 grams, only 150 grams of $KHCO_3$ are weighed out and 101.4 grams of K_2CO_3. Dissolve in water at 50° C. make up to 600 cc.; add 6.928 grams of copper sulphate in 100 cc.; add the alkaline fluid and make up to a liter. To remove lime, which must be done to insure success, the author replaces sodium carbonate, which influences the result, by a mixture of bicarbonate and oxalate of sodium. He proceeds as follows: Dissolve 20 grams of the substance to be analyzed in about 140 cc. of water; add, according to the nature of the material, 10 to 30 cc. of the sodic solution, bring to a boil, cool, and make up to 200 cc. Shake with 1 to 4 grams of animal black; shake, filter, add 5 cc. of this boiling solution to 100 cc. of the reagent, and proceed as usual.

Rev. internat. falsificat., **7**, 46, 47. F.

190. Baumann, J., 1892.—*A new constant for the determination of invert sugar by Herzfeld's method* (*Beitrag zur Invertzuckerbestimmung mittels Fehling'scher Lösung*).

It is not always possible to use 10 grams of material, nor is it correct to multiply by 2 the figures obtained from the use of 5 grams. A new table has been made out, therefore, using the following formula, in which x = copper obtained. Invert sugar $=0.3164+0.010054x+0.000003021x^2$.

Ztschr. Ver. Rübenzucker-Ind., (1892) **42**, 824; Agric. Science, **7**, 185.

W. E. S.

II.—Volumetric estimation.

191. Gerrard, A. W.—*New method for the estimation of grape sugar.*

The author uses three solutions: (1) Copper sulphate, 69.3 grams in 500 cc.; (2) Rochelle salt, cryst., 175 grams, and sodium hydroxid, 76.56 grams in 500 cc.; (3) potassium cyanid, 33 grams to 500 cc. Five cubic centimeters of each are added to 50 cc. of water, brought to a boil, and the grape sugar added until the blue color is discharged. The glucose value is approximately half that of ordinary Fehling's solution.

Pharm. J. Trans. (3), **52**, 208–210; Pharm. Rundschau; Jour. Chem. Soc., **64**, ii, 248, 249; Chem. Centralbl., 1893, i, 445. J. W. F.

192. Samelson, May 1, 1894.—*The volumetric estimation of sugar* (*Ueber die Maassanalytische Zuckerbestimmungsmethode*).

The author holds that Volumetric methods for the estimation of sugar may be just as accurate as Gravimetric methods, when properly conducted. He states that it is only necessary that the work be carefully done, and calls especial attention to the necessity of determining accurately the strength of the copper sulphate solution.

Ztschr. angew. Chem., 1894, 267; Chem. Centralbl., 1894, i, 1102. W. D. B.

193. Bornträger, A.—*Control tests of Fehling's solution.*

For the preparation of an invert-sugar solution to use in determining the *titre* of Fehling's solution, the author recommends that the inversion be effected in the cold. Nineteen grams of sucrose are dissolved in water; 10 cc. of hydrochloric acid of 1.188 sp. gr. are then added and the solution made up to 100 cc. The solution is allowed to stand over night, and 25 cc. are then measured out, neutralized with alkali, and the liquid made up to a volume of 1,000 cc. The solution, thus prepared, contains 0.5 gram sucrose per 100 cc. of liquid. The sucrose used in the preparation of the invert sugar is prepared by purifying refined sugar by treatment with alcohol.

Bull. Assoc. Chim. France, **11**, 564; Ztschr. angew. Chem., 1893, 600; Ber. chem. Ges., **26**, R, 1020, 1021; Vjschr. Chem. Nahr., **8**, 237. B. B. R.

III.—Saccharimetric coefficient.

194. Grimbert, L., 1892.—*A new saccharimetric coefficient for dextrose.*

With regard to the saccharimetric coefficient for glucose, the suggestion is made of a new constant, by which each degree of the saccharimeter should represent not 3.20 grams of anhydrous dextrose per liter, as now, since this is based on the specific rotation of dextrose calculated to the anhydrous material; for 2.06 grams of water-free dextrose per liter is said to give a rotation of 1°, and therefore to be the true saccharimetric constant.

Jour. prakt. Chem. (5), **26**, 253; Jour. pharm. chim., 1892, Sept.; Agri. Science, **7**, 184; Bull. Assoc. Chim. (3), **10**, 598. W. E. L.

195. Sidersky, D., March, 1893.—*The value of the saccharimetric coefficient of dextrose (Sur la valeur du coefficient saccharimétrique du glucose).*

Referring to the article of Grimbert with regard to the value of the saccharimetric coefficient of dextrose, the author states that the figure calculated by him from the specific rotary power given by Tollens, Landolt, and others is 2.032 grams per liter for each division of the saccharimeter scale. The ratio of the rotary power of dextrose to that of sucrose calculated from the same data is 79.1:100 for a 5 per cent, while the saccharimetric coefficient is given as 20.48, or equal to 2.048 grams per liter. The author disclaims any knowledge of the origin of the ratio of 73:100.

Bull. Assoc. Chim. France, **10**, 658. B. B. R.

IV.—Effect of alkalis and barium upon.

196. Beaudet, L., January, 1893.—*The effect of alkalis upon dextrose and levulose (Des transformations du glucose et de la lévulose en présence des alcalis).*

The results of a number of experiments with regard to the influence of alkalis and alkaline earths upon dextrose and levulose are reported, baryta, lime, and sodium carbonate being employed in making the tests. The author concludes that glucose (dextrose) and levulose are not precipitated by baryta in either hot or cold solutions.

The experiments reported show, it is claimed, that dextrose and levulose are destroyed by baryta, lime, and sodium carbonate under the influence of heat, the duration of the heating and the amount of alkali employed being factors in the effects produced.

Bull. Assoc. Chim. France, **10**, 509. B. B. R.

197. Beaufret, January, 1893.—*The precipitation of reducing sugars by baryta (De la précipitation des sucres réducteurs par la baryte).*

In referring to the articles of Beaudet and Vivien, the author states that his experience has shown that reducing sugars are partially destroyed and partially precipitated by baryta.

Bull. Assoc. Chim. France, **10**, 500. B. B. R.

198. COURTONNE, H., February, 1893.—*The action of baryta upon dextrose and levulose* (*Action de la baryte sur le glucose et sur le lévulose*).

In referring to the results reported by Beaudet and Beaufret, the author states that, in experiments made by him, a solution of 17 grams of invert sugar in 100 cc. water, in the presence of an equivalent of barium or strontium hydrate, at about 70° gave an abundant precipitate, which, however, disappeared in a very short time. This solution exercised no reducing influence on a cupric solution, nor did carbon, dioxid give a precipitate with it. This indicates the complete destruction of invert sugar under these conditions.

Bull. Assoc. Chim. France, **10,** 564. B. B. R.

5. Cane sugar.

I.—SOLUBILITY.

199. HERZFELD, A., May 25, 1892.—*Solubility of sugars* (*Ueber das Ergebniss der Versuche betr. Löslichkeit des Zuckers*).

The author gives the conclusions drawn from the results of numerous experiments with regard to the solubility of sugars in pure water and also in water containing both organic and inorganic salts in solution. The results and conclusions differ materially from those of Marschall. It is found in general that the proportion of sugar capable of being held in solution in impure sugar solutions is less than in pure water.

Ztschr. Ver. Rübenzucker-Ind., **42,** 562. B. B. R.

200. HERZFELD, A., 1892.—*The solubility of sugar* (*Ueber das fernere Ergebniss der Versuche betr. die Löslichkeit des Zuckers*).

The author gives further and more detailed results of experiments upon the solubility of sugar, both in pure water and in water containing salts in solution. A table is also given, showing proportions of sugar soluble in water at temperatures from 0° to 100° C., and also the specific gravities and degrees Brix, both at the temperature at which the solution was effected, and also at 17.5° C., the normal temperature for sugar work.

Ztschr. Ver. Rübenzucker-Ind., **42,** 763. B. B. R.

II.—BOILING POINT OF SOLUTION.

201. CLAASSEN, H.—*Boiling points of sugar solutions.*

A table is presented which gives the boiling points of pure sugar solutions under the normal pressure, the densities of the solutions ranging from 10° to 85° Brix. The figures agree closely with those secured by Flourens.

Ztschr. Ver. Rübenzucker-Ind., 1893, 267; Bull. Assoc. Chim. France, **11,** 45.
B. B. R.

III.—CONGELATION POINT OF SOLUTIONS.

202. TRAUBE.—*Point of congelation of sugar solutions* (*Point de congélation des solutions sucrées*).

The points of congelation of sugar solutions for varying degrees of concentration are given by the author. The temperatures at which congelation takes place vary from 3.3° C. for 50.418 grams of sugar per 100 of water, to 0.04° C. for 0.372 gram of sugar per 100 grams of water.

Jahresber. Stammer, 1892, 84; Bull. Assoc. Chim. France, **10,** 385.

IV.—*a*-NAPHTHOL TEST FOR CANE SUGAR.

203. RAPP, G., and BESEMFELDER, F.—*The detection of sugar by means of a-naphthol.*

In testing for sugar by means of this reagent the authors employ a test tube of special construction, provided with a small tube passing through the side of the test tube proper and reaching nearly to the bottom of the latter. In addition a small funnel is fastened in the neck of the test tube, and in this 2 or 3 drops of a 20 per cent alcoholic solution of α-naphthol are placed. The liquid to be examined is next poured in until the tube is about one-fourth full. Sulphuric acid is now run in through the small tube previously referred to, and if sugar is present a violet zone is observed at the line of contact of the two liquids, which spreads by diffusion. One part of sugar per 10,000,000 yields a pale lilac coloration, while with more than 0.2 per cent sugar present the liquid blackens at once.

D. Zuckerind., 1892, 538; Jour. Soc. Chem. Ind., **12**, 183. B. B. R.

V.—FERMENTATION PRODUCTS.

204. O'SULLIVAN, J.—*The action of yeast on cane sugar.*

A series of experiments are described which show that the whole or a large proportion of cane sugar in solution may be inverted by yeast, while either very little, or none whatever, is fermented. Tests with regard to the partial fermentation of cane sugar show that in the first stages dextrose is fermented in larger proportions than levulose. The concentration does not influence the amount of sugar inverted in a given time with the same quantity of yeast.

Tr. Inst. Brewing, **6**, 67; Jour. Soc. Chem. Ind., **12**, 282. B. B. R.

205. O'SULLIVAN, J., 1892.—*The hydrolytic functions of yeast.*

The author proposes to show: (1) "That healthy yeast yields none of its invertase or hydrolytic power to water in which it is washed; (2) that when healthy yeast is placed in contact with a cane-sugar solution, the hydrolysis that takes place is solely an action 'under the immediate influence of the plasma of the cell,' and that no invertase leaves the yeast cell during the time that the hydrolysis is being effected."

Healthy brewer's yeast was prepared by washing well with distilled water and filtering through linen. Some difficulty was experienced in filtering off the yeast cells so that the solution could be tested for hydrolytic power. The pulp of Swedish filter paper was found to be effectual in catching up the yeast cells, while it had no influence upon the hydrolytic action of invertase. By treating cane-sugar solutions of different strengths with various amounts of the healthy yeast as prepared above, and of the washings of the yeast, it was found that while the solutions treated with yeast changed in their opticities, those treated with the washings of the yeast did not, even after standing forty-eight hours. The latter solutions were subsequently inoculated with a small amount of invertase solution, and after twenty-four hours the opticities of the solutions were uniformly affected, showing that they were capable of being inverted if only a trace of invertase was present.

A number of experiments with cane sugar in which, after treating with yeast for a short time, the yeast was removed from a portion of the solution and the opticities of each immediately tested, then set aside for some time and again tested, showed that no inversion was effected after the removal of the yeast cells from the solution, while the other portion continued to change its opticity. In addition to the above, the number of yeast cells were counted in a number of the experiments, and at various stages, and the author arrives at the conclusion that—

"The hydrolytic action of yeast on cane sugar takes place without increase of yeast, and there is no alcohol formed.

"The hydrolysis of cane sugar under the influence of yeast is a case of zymic hydrolysis," as well as those stated above.

Jour. Chem. Soc., **61**, 593–605, 926–943. J. W. F.

VI.—ESTIMATION OF SUCROSE, DEXTROSE, AND LEVULOSE.

206. WIECHMANN, F. G., 1892.—*Determination of sucrose, dextrose, and levulose mixture.*

A solution is carefully prepared and its density and rotatory power determined; in a weighed portion the amount of reducing sugar is gravimetrically determined with Fehling's solution; another weighed portion is inverted and the total reducing sugar in it is determined. From algebraic formulæ the amount of the several sugars can then be determined. See original for details.

School of Mines Quarterly, **13,** No. 3; Chem. News, **66,** 237–239, 249–251; Agric. Science, **7,** 185; Jour. Chem. Soc., **64,** ii, 99. W. E. S.

VII.—ESTIMATION OF RAFFINOSE.

207. LINTNER and DÜLL, 1892.—*Raffinose determination.*

Raffinose does not occur in the sugar beet, and so does not interfere with sucrose determination there. The only reliable method for the determination of raffinose in sugar products is that of Clerget as modified by Herzfeld.

Ztschr. ges. Brauwesen, **15,** 145; Agric. Science, **7,** 183. W. E. S.

VIII.—ESTIMATION OF SUCROSE AND RAFFINOSE.

208. MEHAY, 1892.—*Determination of sucrose and raffinose in mixture.*

The author proposes the following formulæ for this purpose: $S = K_1A + K_2B$, and $R = K_3A - K_4B$, in which A and B are the direct and indirect polarizations of normal weights and K_1, K_2, K_3, and K_4 are temperature coefficients.

Sucr. indig.; Chem. Centralbl., 1892, **1,** 508; Agric. Science, **7,** 185.

209. WEISBERG, J., November, 1892.—*Sucrate and raffinosate of lead* (*Saccharosate et raffinosate de plomb*).

The trisaccharosate of lead formed on the addition of ammoniacal lead subacetate to an aqueous sugar solution, is insoluble in water and in alcohol, but soluble in sugar solutions. Upon this property Lopez has proposed to base a method for the separation of sucrose from raffinose. The experience of the author is that raffinose precipitated by an excess of ammoniacal lead subacetate in an aqueous raffinose solution is also soluble in aqueous sugar solutions.

Bull. Assoc. Chim. France, **10,** 434. B. B. R.

6. *Special sugar products.*

I.—ESTIMATION OF JUICE AND SOLIDS IN RAW PRODUCTS.

210. VIVIEN, A., May, 1893.—*Determination of the amount of juice in the beet* (*Détermination de la quantité de jus dans la betterave*).

The results of the determination of the amount of juice in the beet by the different processes in general use are reported, and the relative merits of the several methods are compared. The processes commonly employed are: (1) The direct determination of the sugar in the beet and also the sugar in the juice; (2) the determination of water both in the beet and in the juice; (3) washing thoroughly with water the beet pulp, and weighing the residue after drying.

The first method is inexact and its use inadvisable on account of the fact that the sugar content of the juice obtained by expression and that by extraction are not the same, while the second process is quite tedious and little employed. The last-mentioned method gives more exact results than either of the others and is more generally followed. The author in executing this process washes the beet pulp by decantation with water of gradually increasing temperature until 80° C. is reached, the insoluble residue being finally brought upon a tared filter. A table based upon the results of numerous determinations of this character is given, the proportions of juice in the beet corresponding to different densities being shown.

Bull. Assoc. Chim. France, **10,** 833. B. B. R.

211. GIESBERS, M. E., March, 1893.—*Determination of the amount of juice in sugar cane.*

In estimating the proportion of juice in cane the author proposes to determine the amount of water, both in the cane and in the juice, and then to calculate the juice content by the following formula: $\frac{W}{W'} \times 100 =$ per cent of juice; in this formula W

is the per cent of water contained in the cane and W' the amount in the juice. In determining the water content of the cane, the finely divided sample was dried at not exceeding 100°, while the water percentage was likewise determined in the juice expressed from portions of the cane taken from the same parts of the stalk from which samples had been taken for the former estimation. By this process a juice content of 88.03 per cent was secured for one sample, as against 88.08 per cent obtained by washing out soluble matters from the cane with hot water, drying the residue, and subtracting its weight from 100.

La. Planter, **10**, 157. B. B. R.

212. Pellet, H., June, 1893.—*Determination of amount of marc and juice in beets (Détermination de la quantité de jus contenu dans la betterave).*

Reference is made to the process recommended by Vivien, in which water at 80° C. is employed in washing the beet pulp in order to extract soluble matters. The author claims that water of this temperature dissolves out the pectic matters in greater or less proportions, the results varying with the duration of the washing and the amount of water employed. The employment of the cold aqueous process is recommended, therefore, for the determination of marc, the pulp used being, preferably, in a very finely divided condition.

Bull. Assoc. Chim. France, **10**, 948. B. B. R.

II.—METHODS OF EXTRACTION FOR ANALYSIS.

213. Pellet H., January, 1893.—*Beet analysis by the aqueous method (L'analyse de la betterave par la méthode aqueuse).*

The author, in referring to the modification of the aqueous method proposed by Lewenberg, states that the process is made more tedious and complicated by the measurement of the water and lead subacetate from burettes, as is required by this modification. The amount of lead acetate (3 cc.) employed is, moreover, too small in many cases. Instead of adopting 23.2 cc. as the volume occupied by the normal weight of pulp, the author employs the weight of 25.87 grams of pulp in order to make compensation for the volume occupied by the marc, the total volume being made up to 200 cc. and the solution polarized in a 400 mm. tube. The unsatisfactory results reported from the use of the cold aqueous method are due to failure to comply strictly with the proper directions for its execution. By adherence to the proper precautions no trouble from foaming has been experienced.

Bull. Assoc. Chim. France, **10**, 519. B. B. R.

214. Dupont, F.—*Official analysis of beets in Belgium.*

The alcoholic extraction process is recommended as being the most accurate and satisfactory method for sucrose determinations in the beet, viewed from a scientific standpoint. The alcoholic digestion process, although inferior to the above process as regards its scientific value, is considered as sufficiently exact for ordinary work and at the same time easy of execution. The hot aqueous digestion method, though not equal in scientific importance to the alcoholic extraction process, is easily carried out and when executed under proper precautions gives results sufficiently exact for all practical purposes. The cold aqueous digestion process, when all necessary conditions are complied with, gives entirely trustworthy results, but in actual practice it is difficult to so carry out all the essential details as to insure absolute accuracy.

Bull. Assoc. Chim., **11**, 317. B. B. R.

215. Pellet, H., June, 1893.—*The aqueous method applied to cane analysis (Dosage direct du sucre dans la canne).*

The finely cut material is brought into a flask, water is added, then 5 or 6 cc. lead subacetate solution, and the whole heated for half an hour on a boiling water bath. After cooling, the liquid is diluted to a definite volume, shaken, filtered and polarized in a 400 mm. tube. To compensate for the volume occupied by the marc (or fiber), instead of diluting to 200 cc., the volume should be made up to 201.6 or 202.6

cc., according to the normal weight employed. If a 200 cc. flask is employed, proper correction can be made by weighing 16.07 grams or 25.71 grams of the sample, instead of the regular normal weight.

Bull. Assoc. Chim., France, **10**, 924. B. B. R.

216. MASSON.—*Pellet's cold aqueous digestion process* (*Rübenanalyse durch Pellet's kalte wässrige Digestion*).

It is noted that this method requires careful working by practiced chemists, and, very often, control by the alcoholic digestion, which alone is considered scientifically exact: otherwise great differences are unavoidable. One considerable objection is that air remains retained in the interstices of the pulp, occupying a considerable volume and thus greatly affecting results.

As Sachs has shown, it is necessary to prepare the pulp from all portions of the beet, since in this way only can a uniform sample be obtained. The method is considered not at all adapted to practical work, and the results secured are untrustworthy.

Bull Assoc. Chim., Belge., **6**, 210; Chem. Ztg. Rep., **17**, 123. B. B. R.

217. SACHS.—*Pellet's cold digestion process* (*Pellet's kaltes Digestionsverfahren*).

This process, which obtained general introduction on account of simplicity and cheapness, as well as exclusion of the possibility of indicating too much sugar, is pronounced to be entirely unsatisfactory, in view of numerous tests which have been recently made. A large number of experiments have shown that this method yields higher results than the alcoholic and hot water digestion processes, these differences being as much as 0.5 per cent in some cases.

Sucr. Belge., **21**, 127; Chem. Ztg. Rep., **16**, 366. B. B. R.

218. BODRI, A.—*The direct method of beet analysis* (*Ueber die Anwendbarkeit der direkten Methoden der Rübenanalyse*).

After reviewing the result of a series of experiments in which the relative merits of the methods of Pellet and Scheibler, and the cold and the hot alcoholic digestion processes are compared, the author concludes that the cold aqueous digestion method is the most satisfactory. The ease of execution of this methed and the little time consumed, as compared with the alcohol extraction where the amount of time employed affects the results considerably and where the greatest care in the hands of experienced workers is required, renders the former more desirable for practical work.

Zapiski, 1892, 23; Böhm. Ztschr. Zuckerind., **17**, 264. B. B. R.

219. LEBORGNE, February, 1893.—*Pellet's cold aqueous digestion process* (*La digestion aqueuse à froid*).

The results of a large number of comparative tests of the alcoholic and the hot and cold aqueous digestion processes are reported, the figures obtained by the three processes being quite concordant, both for individual samples and for the averages of all the determinations. By the use of the cold aqueous process in conjunction with the continuous polarization tube of Pellet the sucrose determinations can be executed both expeditiously and accurately.

Jour. fabr. sucr., **34**, No. 6. B. B. R.

220. WEISBERG, J.—*The aqueous digestion process* (*La digestion aqueuse à chaud et la digestion aqueuse à froid*).

The author calls attention to the necessity of employing an excess of lead subacetate in the hot aqueous digestion process, the lead solution being added before the commencement of the heating. Five cubic centimeters of the subacetate solution (27 or 28° Beaumé) are generally sufficient for the normal weight of the beet pulp· Sidersky has claimed that in the hot aqueous process pectic matters go into solution, but the experience of the author is to the effect that the pectic matters which come into solution are precipitated in the presence of an excess of lead subacetate.

Jour. fabr., sucr., **34**, No. 4. B. B. R.

221. WEISBERG, J., November, 1892.—*The cold aqueous process for the analysis of diffusion chips* (*Le dosage du sucre dans les cossettes de betteraves par la digestion aqueuse à froid*).

The author calls attention to the necessity and importance of the beet pulp being in a sufficiently finely divided condition in order to secure the best results by the cold aqueous digestion process. In order to obtain a pulp of a sufficient degree of fineness, it is recommended that an apparatus like that devised by Kiehle be employed, since by its use a practically impalpable mass can be secured from the diffusion chips.

Sucr. indig., **40**, 535. B. B. R.

222. WEISBERG, J., December, 1892.—*Modification of the aqueous method for beet analysis* (*Modification au dosage du sucre dans la betterave par la methode aqueuse*).

The author refers to a modification of the aqueous method of sugar determination recently proposed by Lewenberg. The two chief objections of the latter to the regular aqueous method are that difficulty is experienced in correct gauging in the globular flask on account of great foaming, and that there is some question as to the volume occupied by the beet pulp. As the result of his investigations, he concludes that, on an average, 23.2 cc. represents the volume of 26.048 grams of pulp. In the process proposed, 26.048 grams of pulp are brought into a glass vessel, and 73.8 cc. distilled water are run in from a burette; 3 cc. lead subacetate are then added, thus completing the volume of 100 cc., and after agitation for ten minutes the liquid is filtered and polarized. Weisberg observes that Lewenberg is not the first to recommend this mode of procedure, Kayser having employed it previously, using the figure 24 cc. as equivalent to 26.048 grams of pulp.

Bull. Assoc. Chim. France, **10**, 472. B. B. R.

223. WEISBERG, J., May, 1893.—*Determination of sugar in the beet* (*Dosage du sucre dans la betterave par les méthodes alcoholiques et aqueuses*).

Herzfeld has raised an objection to the hot aqueous digestion process on the ground that the lead forms soluble compounds with the pectic materials of the beet, giving rise to higher polariscopic readings on account of the dextrorotary tendency of the compounds formed. The author reports the result of tests of the hot aqueous methods as compared with the alcoholic process, tests of the former method being also made in the presence of a considerable proportion of pectic materials which were purposely added.

The results of all these experiments show a close agreement and indicate the utility of the hot aqueous method, even where pectic bodies are present.

Bull. Assoc. Chim. France, **10**, 840. B. B. R.

224. MARONIER, A., December, 1892.—*Determination of sugar in bagasse* (*Dosage du sucre dans la bagasse*).

Of the finely cut material 100 grams are weighed into a porcelain dish, hot water is added and 0.05 gram of an aqueous solution of lime. The mixture is heated an hour and a half at a temperature not exceeding 90° C., and after allowing to cool the lime is neutralized and a sufficient amount of water is added to bring the total weight to 1,300 grams. After the wet bagasse is pressed, 100 cc. of the aqueous solution obtained by digestion is placed in a flask, 4 cc. lead subacetate added, and then water run in up to the 110 cc. mark. After filtration the liquid is polarized in the usual manner.

Bull. Assoc. Chim. France, **10**, 479. B. B. R.

225. CLAASSEN.—*The influence of the fineness of beet pulp upon the composition of the juice* (*Einfluss der Feinheit des Rübenbreies auf die Zusammensetzung des Saftes*).

To test the influence of fineness of division of beet pulp upon the composition of the juice extracted by expression a large number of beets were cut into halves; one half of each beet was very finely divided, while the pulp obtained from the other half was very coarse. Both the coarse and the finely divided pulp were pressed under the same conditions and the juice from each polarized. The sugar content of the coarse pulp was also determined by extraction. The figures obtained for the juice from the fine pulp were uniformly much higher than for the coarsely divided substance, and the latter material also yielded a higher sugar content by extraction than by expression. These experiments, it is claimed, show the necessity of abandoning the hitherto employed methods of juice examination and giving more attention to extraction.

D. Zuckerind, 266; Böhm. Ztschr. Zuckerind., **17**, 264. B. B. R.

III.—TESTING OF SEED BEETS.

226. HERLES.—*The examination of beets for seed* (*Neues Untersuchungsverfahren zur Auswahl der Samenrüben*).

It is recommended that instead of employing the polariscope for beet testing that the samples be finely cut, the juice expressed, and the density determined by means of a pycnometer, employing a specially constructed balance, which gives at once the proper density of reading. It is claimed that this is better adapted to determining the value of beets than by employing Pellet's cold digestion process, which latter method in many factories gives rise to differences in results, which are due largely to differences in fineness of the pulp, temperature of the water, time consumed, etc.

Böhm. Ztschr. Zuckerind., **17**, 91; Chem. Ztg. Rep., **16**, 365. B. B. R.

227. DÖRSTLING.—*Herles' method of beet analysis* (*Herles' Methode der Mutterrüben-Analyse*).

The method proposed by Herles for selection of beet seed by means of the specific gravity of the juice has been shown to be unsatisfactory by numerous experiments. The cold aqueous digestion method, employing continuous polarization tubes, is unconditionally to be preferred.

D. Zuckerind., **18**, 124; Chem. Ztg. Rep., **17**, 16. B. B. R.

228. COURTONNE, H.—*Sampling and preservation of beet juice.*

For the preservation of beet-juice samples for analysis the author recommends the employment of an alcoholic solution of mercuric chlorid (0.2 per cent). Juice thus treated can be used for all ordinary determinations, corrections both for increase in volume and solids being made in density and solids determinations, while for sucrose and ash estimations a correction for the dilution alone will be necessary.

Bull. Assoc. Chim. France, **11**, 316. B. B. R.

229. HERLES.—*Preservation of sugar juices* (*Conservirung der Zuckersäften zu analytischen Zwecken*).

It is recommended that to one liter of juice there be added 10 cc. of carbon disulphid or chloroform, the latter being preferable; when pure and acid-free it keeps the thin juice clear and unchanged even for as long a period as a year.

Oesterr. Ztschr. Zuckerind., **21**, 763; Chem. Ztg. Rep., **16**, 365. B. B. R.

IV.—CORRECTION FOR SUSPENDED SOLIDS.

230. LENOBLE, E., April 4, 1894.—*Correction to be applied in the determination of the strength of a solution containing a solid in suspension* (*Correction dans la détermination du titre d'une liqueur tenant en suspension un précipité. Application à la saccharimétrie*).

Two methods are suggested, both of which are modifications of, and less convenient than, Scheibler's double dilution method.

Bull. Soc. Chim. France, 1894 [3], **11**, 336. W. D. B.

V.—ESTIMATION OF INVERT SUGAR IN BEETS.

231. CLAASSEN, H., March, 1893.—*Determination of reducing sugars in the beet* (*Le sucre réducteur dans les betteraves et les jus de diffusion*).

The necessity of determining the reducing sugars by a direct process and not in the juice obtained by expression has previously been noted by the author, the hot aqueous digestion process being preferable for the extraction of these sugars from the beet. In this process an excess of lead subacetate is to be avoided on account of the destruction of reducing sugars thereby occasioned; it is also necessary to neutralize the acids of the juice in order to prevent inversion of sucrose. To 110 grams of finely divided chips, 10 to 15 cc. of lead subacetate are added and also 1 to 2 grams of precipitated carbonate of lime; the whole is then digested in a half liter flask for three-fourths hour to one hour on a water bath. After cooling and completing the volume with water, 100 cc. of the decanted liquid is clarified with a little lead subacetate and the volume made up to 110 cc. To 100 cc. of the filtrate from this solution, sodium carbonate solution is added to precipitate the excess of lead, the volume being raised to 200 cc. After filtering, 100 cc. is employed for the determination of reducing sugars by the usual process.

Sucr. Indig., **41,** 242; D. Zuckerind., **17,** 1372; Chem. Zeitg. Rep., **16,** 295.

B. B. R.

VI.—ESTIMATION OF SUCROSE IN BEETS.

232. PELLET, H.—*Determination of sucrose in the beet.*

A number of experiments were conducted by the author with reference to the influence of the state of division of the beet pulp (or chips) upon the rate of diffusion of the sugar contained therein. The experiments were made in connection with the cold aqueous digestion process, and the results secured showed the importance of a fine state of division of particles in order to obtain a quick and satisfactory extraction. A number of tests were also made to determine the influence of the presence or absence of subacetate of lead upon the rate of diffusion. Where tests were made immediately after preparation of the solutions, the rate of diffusion, with and without lead subacetate, was practically the same, but after a somewhat prolonged digestion, the coefficient of diffusion was somewhat higher in solutions containing no subacetate. Experiments are also reported, which were designed to test the influence of temperature upon the extraction of sugar from the beet pulp. It was found that at 80° C. a digestion of one-fourth hour gave practically as complete an extraction as one-half hour at 50° C. Comparatively satisfactory results can be secured by employing water at 80° to 85° in making up the solutions, and allowing the hot solution to cool slowly. The use of the water bath can thus be dispensed with.

Bull. Assoc. Chim. France, **11,** 255. B. B. R.

233. PELLET, H. September 5, 1893.—*Methods of determining sugar in beets* (*Dosage du sucre dans la betterave. Méthodes anciennes et nouvelles. Sélection des betteraves porte-graines*).

A description of the principal methods which have been or are used for the estimation of sugar in the sugar beet.

Bull. Soc. Chim., 1893 [3], **9,** 735–740. W. D. B.

234. PELLET, H. 1894.—*Determination of the crystallizable sugar in the beet.*

The author suggests a polariscopic method where water is used as the solvent for extracting sugar instead of alcohol. It is known as the method of instantaneous aqueous diffusion in the cold. A suitable amount of the pulp, obtained in a very finely divided state, is weighed into a flask of definite volume. If the normal weight for the German instruments, 26.048 grams, is used, the volume should be 201.35 cc., or if 25.87 grams be taken, the volume should be 200 cc. The pulp is passed into the special flask with the assistance of a jet of water, then 5 to 7 cc. of subacetate of lead (30° B.) are added and a little ether to reduce the foam, the flask being agitated at the same time. Make up to 201.35 or 200 cc., as the case may be, and mix thoroughly. Filter and polarize after having acidulated the liquid with one or two drops of glacial acetic acid. A 400 mm. tube is used for the observation and gives the reading directly, corrected for the increase in volume of the solution. The diffusion is instantaneous and complete, permitting the filtration to be made as soon as the flask has been filled to the graduation and the contents thoroughly mixed. It is unnecessary to heat the solution, unless the water is extremely cold, approximating 0° C.

Jour. Am. Chem. Soc., **16**, 266–273. J. P. S.

VII.—WATER IN RAW SUGARS.

235. HERZFELD.—*Water estimation in raw sugars* (*Wasserbestimmung im Rohzucker*).

The usual time of drying of four hours is not always sufficient, but six hours should be adequate for all cases. In practice an absolutely constant weight is not readily to be attained and requires even too long a time *in vacuo*. The vacuum apparatus is not considered necessary for general use, but on account of quicker drying and greater accuracy in case of acid and inverted products, it is to be much commended. The old theory that sugar products, by long drying, absorb oxygen from the air, giving an increase in weight, was not confirmed either for pure or raw sugar, even after fifty-six hours drying.

Ztschr. Zuckerind., **43**, 130; Chem. Ztg. Rep., **17**, 66; Bull. Assoc. Chim. Belg. **6**, 268.

236. SACHS, F.—*Water determination in raw sugars.*

The author confirms the results of Herzfeld's latest investigations with regard to the estimation of water in sugars. A drying *in vacuo* is unnecessary so long as one has at hand a good drying chamber, with which a constant temperature of 107° (not lower at any rate than 105°), can be secured. Under 105°, correct results can not be obtained, since the water of crystallization and combined water can not be completely expelled below that temperature.

Bull. Assoc. Chim. Belge., **6**, 267; Chem. Ztg. Rep., **17**, 134. B. B. R.

237. SACHS.—*Water determination in raw sugars and masse cuite* (*Die Wasserbestimmung in Rohzuckern und Füllmassen*).

Numerous concordant experiments show that with raw sugars, with three to four hours' heating, in a drying chamber of ample size and double walls, at a temperature of 107° C., the best results are secured; more quickly, in fact, than with a vacuum apparatus at 100°. For masse cuites, 26.048 grams are dissolved and made up to 100 cc.; 5 cc. of the solution are mixed with 7 grams of sand, or 1 to 1.5 grams of more porous silica and dried five hours at 107°.

Bull. Ass. Chim. Belge. **6**, 221; Chem. Ztg. Rep., **17**, 123. B. B. R.

238. STIFT, A.—*Water determination in raw sugar* (*Wasserbestimmung im Rohzucker*).

The employment of a water oven with a drying temperature of 100° C. is recommended for water estimations in sugars, and where possible the operation should be conducted *in vacuo*. Even at 100° C. marked changes may take place in the composition of sugars, and these changes are in proportion to the amount of organic substances (non-sugars) present; alkaline sugars give off ammonia, while acid products evolve volatile fatty acids. The polarization also diminishes quite appreciably, sometimes as much as 1 per cent.

Wien. Woch., **30**, 405; Chem. Ztg. Rep., **16**, 244. B. B. R.

239. Drenckmann, May 25, 1892—*Water determination in raw sugars* (*Wasserbestimmung bei Rohzucker*).

Among other processes tried, it was found that quite satisfactory results could be secured by using the method employed by the agricultural chemists for drying easily decomposible organic substances. This consists in heating in a bath exactly to 100° C. by means of a salt solution, a current of previously heated hydrogen being at the same time passed through the drying vessel. This process, while very accurate, is entirely too time-consuming for commercial work and a drying apparatus has therefore been devised which permits of direct heating and also of easy regulation of the temperature. This drying chamber permits of the drying of a large number of samples at one time, and in order to prevent the proper temperature being exceeded, the bulb of the thermometer is immersed in paraffin or, better still, in mercury.

Proc. Gen. Versamm. Ver. Rübenzucker-Ind., 1892: Ztschr. Ver. Rübenzucker-Ind., **42**, 612. B. B. R.

240. German committee.—*Water determination in raw sugars* (*Wasserbestimmung bei Rohzucker*).

Resolution adopted by a committee of commercial chemists appointed to consider the question at Berlin, October 18, 1892:

"The water determination should be executed only at a temperature of 105° to 110° C., and there should be employed therefor only that drying chamber, which in its construction offers the guarantee that the temperature indicated by the thermometer prevails."

Ztschr. Ver. Rübenzucker-Ind., **42**, 857. B. B. R.

241. Sterbo, F., May, 1893.—*Water determinations in raw sugars* (*Die Wasserbestimmung im Rohzucker*).

The experiments of Strohmer, Stift, and Herzfeld are referred to, and the author concludes with them that the vacuum drying apparatus conforms to most of the requirements of commercial analysts, yielding both quick and exact results. A new apparatus is described which combines the principle of the vacuum with that of Soxhlet's apparatus. The drying chamber proposed has double walls, and its interior is connected with a vacuum pump. Dry air of 105° C. is conveyed through the inner chamber, which contains the sample, by means of suction from the pump, the rate of influx of the air being so regulated that a pressure equal to only 25 cm. of mercury is maintained in the chamber. Highly satisfactory results can be secured with this apparatus, first products requiring only from two to three hours for complete drying.

Chem. Ztg., **17**, 757. B. B. R.

242. Herles.—*Water determination in masse cuite* (*Wasserbestimmung in Zuckerfüllmasse* .

The drying of masse cuite is rendered easier when it is mixed with an indifferent, easily volatile liquid, which, however, does not dissolve the grain. The mixture with sand can also be made more readily and more uniformly.

The employment of methyl alcohol is recommended for this purpose, 10 cc. being added to 4 or 5 grams of masse cuite along with 25 or 30 grams of sand. This mass is warmed for two minutes in the drying chamber, mixed well and dried in the usual manner; first, for one hour at 80° or 90° and gradually raising the temperature to 105° C. Satisfactory results can be secured in four hours, and it is claimed that the method is also applicable to sirups and molasses.

Oesterr.-ungar. Ztschr. Zuckerind., **21**, 764; Chem. Ztg. Rep., **16**, 366. B. B. R.

243. Josse, A., March, 1893.—*Determination of water in sirups and masse cuite* (*Dosage de l'eau dans les sirops et les masses cuites*).

The process proposed provides for the employment of a spiral of filter paper, previously dried and weighed, which has a surface of about 100 sq. cm. About 2 grams of the sample are weighed into a capsule, dissolved in about 6 to 8 cc. of water by the aid of heat, the spiral of paper being thereupon placed in the dish and allowed to take up the liquid contents by capillarity. The drying is conducted in a chamber with a temperature of from 100° to 110° C., a constant weight being readily attainable. The process is especially adapted to water determinations in those substances which are ordinarily dried with great difficulty.

[NOTE BY ABSTRACTOR.—This process is essentially the same as that proposed by Dr. Wiley several years since, though the author makes no reference whatever to that fact.]

Bull. Assoc. Chim. France, **10**, 656. B. B. R.

244. BAUMANN, May 25, 1892.—*Water determination in sugars, etc.*

With sirups containing invert sugar, higher results are obtained in the association laboratory in the air bath than *in vacuo*, though quicker and more reliable results are secured by the latter process. The vacuum is only considered necessary for products containing invert sugar, or having a somewhat acid reaction. For ordinary work there can be employed advantageously a double-walled air bath, with direct heating, an asbestos plate being placed upon the shelf of the drying chamber·

Proc. Gen.-Versamm. Ver. Rübenzucker-Ind., 1892; Ztschr. Ver. Rübenzucker-Ind., **42**, 615. B. B. R.

245. WEISBERG, J.—*Determination of dry matter in sugar-house products.*

The author calls attention to the amount of time consumed in determining the amount of dry matter in sugar products, the desiccation frequently requiring eight to twelve hours, even where the material has been mixed with sand. As a substitute for the sand it is recommended that pumice stone, previously treated with sulphuric acid, washed, dried, and ignited, be employed. This material is of quite an inert character and very porous, and when mixed with a sugar product, the drying of the latter can be accomplished rapidly and without any danger of decomposition of any of its constituents.

Bull. Assoc. Chim. France, **11**, 524. B. B. R.

246. COGER. G.—*Determination of water in sugarhouse products.*

In order to insure correctness in the calculated coefficients of purity of the various products of the sugar factory, it is necessary to determine directly the proportion of solid matter rather than estimate it from density. The author describes a specially constructed vacuum drying apparatus devised by him, which permits a rapid and satisfactory estimation of the water content in sugar-house products. Very satisfactory results were secured by the employment of a temperature of from 90° to 95° and a vacuum of from 20 to 22 inches.

Bull. Assoc. Chim. France, **11**, 473. B. B. R.

247. COURTONNE, H.—*The determination of dry matter in sugar products.*

The author refers to the method proposed by Pellet and Terpitz (Bull. Assoc. Chim., September, 1891), for the determination of water in masse cuite, and while the process is commended as being rapid and affording approximate results, it is claimed that the great variability in the composition of products of this class will not permit of the general application of the method, especially if accuracy is desired. The drying of masse cuite in a drying chamber at 105° to 107° C. for thirty hours leads to unreliable results, and the author recommends, in lieu thereof, the employment of the apparatus devised by himself for drying *in vacuo*.

Bull. Assoc. Chim., France, **11**, 262. B. B. R.

248. SEYFFART.—*The estimation of water in raw sugar.*

Böhm. Ztschr. Zuckerind., **43**, 633; Chem. Ztg. Rep., **17**, 219, 220.

VIII.—ESTIMATION OF ASH IN SUGAR PRODUCTS.

249. ALBERTI and HEMPEL.—*Method of determining ash in sugar.*

Jour. fabr. sucr., **32** (1891), 37; Jour. Zuckerind., **16** (1891), 1285; Analyst, **17**, 40. F. W. W.

250. STIFT, A.—*The examination of raw sugars* (*Untersuchung von Rohzuckern*).

The method of ash determination of Alberti and Hempel, as shown by about 600 experiments, is not considered as yet suitable for superseding the sulphate method. With higher grade products the results agree well, but with some sugars, and with masse cuite, molasses, etc., quite considerable differences are observed, these variations amounting to as much as 6 per cent. The sand is attacked by acid products, and this circumstance is of some importance, since sand washed with HCl and ignited is not suitable for further use. It is also found that with different quartz sands the same products show different results.

Oesterr.-ungar. Ztschr. Zuckerind., **21**, 616; Chem .Ztg. Rep., **16**, 277. B. B. R.

251. STIFT, A.—*Alberti and Hempel's quartz-sand method of determining ash* (*Alberti und Hempel's Quarzsand-Veraschungs Methode*).

From a large number of practical tests with various sugar products the conclusion has been reached that this method, by proper execution and by previous testing of the quartz sand (whose bad quality may lead to great errors), yields concordant and satisfactory results. In contrast to other processes, the true content of inorganic substances is obtained, from which the organic nonsugars can be determined. The purity of the quartz sand is one of the chief factors in the process, and uncertainty connected with securing a pure article is an obstacle in the way of the employment of this method. Nitrates in sugar are decomposed in this process, so that a separate nitrate determination is necessary.

Oesterr.-ungar. Ztschr. Zuckerind., **22**, 22; Chem. Ztg. Rep., **17**, 102.

252. COURTONNE, H., July 1892.—*Use of iron oxid for ash determinations of sugar products* (*Incineration des produits sucrés au moyen du sesquioxyde de fer*).

Author finds iron oxid superior to quartz, proposed by Alberti and Hempel, because more rapid, requiring lower temperature, leaving the ash constituents uncombined so that alkalis can be directly leached out and other determinations made, and because platinum vessels are less attacked.

Neue Ztschr. Zuckerrübenind., 1892, **29**, 236, 237; Bull. Assoc. Chim. France, **10**, 223; Centralbl. Agrik. Chem., **22**, 214, 215. W. F.

253. VIVIEN, July, 1892.—*Ash determination by the process of Courtonne* (*Incineration de produits sucrés au moyen du sesquioxyde de fer*).

The results of a number of ash determinations by the ferric oxid method are reported, the tests being executed under varying conditions as regards time and temperature. The ash percentages secured by this method are always lower than those obtained both by the sulphuric acid process and by direct ignition; the results, moreover, by this process are not at all concordant, different figures being obtained for different periods of ignition and different temperatures. By this method it is also impossible to arrive at a point where the ash has a constant composition, and chlorids are attacked and probably partly volatilized. On the contrary, by the sulphate process, a constant weight is easily reached and concordant results are readily secured.

Bull. Assoc. Chim. France, **10**, 225. B. B. R.

IX.—ESTIMATION OF ALKALINITY IN SUGAR PRODUCTS.

254. LACHAUX, L., December, 1892.—*An indicator for tests of alkalinity of juices and sirups* (*Préparation d'un indicateur pour essais de l'alcalinité des jus et sirops en sucrerie*).

Owing to the difficulty of securing quinolin green, the author recommends the following preparation for employment as an indicator for alkalinity determinations in the sugarhouse. Dissolve 3.1 grams of rosolic acid in 150 cc. of alcohol of 90° and make the solution neutral. Dissolve 0.5 gram malachite green in 50 cc. alcohol of 90°. The mixture of these two solutions gives a green liquid, a few drops of which yield a reddish purple with alkaline juices and sirups, turning readily to a green on neutralizing with sulphuric acid.

Bull. Assoc. Chim. France, **10,** 475. B. B. R.

255. Stift, A.—*Alkalinity determinations in sugar products* (*Alkalitätsbestimmung mittels Aether und Rosolsäure*).

The apparently ingenious method of Buisson has proved to be practically of no utility, since the ethereal layer often requires three-fouths of an hour to separate; the results often deviate widely from those obtained by the employment of litmus or phenolphthalein.

Oester.-ungar. Ztschr. Zuckerind., **21,** 662; Chem. Ztg., Rep. **16,** 277. B. B. R.

256. Neumann, K. C., June 20, 1892.—*Determination of alkalinity of molasses* (*Bestimmung der Alkalität der Melasse*).

Litmus solution, which was originally employed as an indicator in tests of this kind, has been superseded to a great extent by phenolphthalein, though with the latter the results secured are frequently too low. The author is of the opinion that an indicator should be employed which will allow the determination of both the free alkali, as well as the alkaline reacting salts, such an indicator being methyl-orange, though this can not be employed for dark-colored solutions. The method of Buisson, employing rosolic acid along with ether, does not yield satisfactory results, nor does the substitution of methyl-orange for rosolic acid suffice. By the employment of litmus tincture, however, in conjunction with ether, the titrations can be conducted in a very satisfactory manner. The molasses is dissolved in water and largely diluted, and after addition of the ether and litmus tincture, is well shaken.

Böhm. Ztschr. Zuckerind., **17,** 22. B. B. R.

X.—Estimation of lime in sugar products.

257. Wolff, J.—*The estimation of calcium salts in sugar juices and sugar products* (*Ueber die Bestimmung der Kalksalze in Zuckersäften und Zuckerproducten*).

Reference is made to the method given years previously by Pellet and also Bittman of the employment of a standard soap solution for the determination of lime, and an improved process based upon the same principle is proposed. The soap solution employed is prepared from pure olive oil, by first forming a lead plaster and then precipitating the lead with potassium carbonate and extracting the mass with alcohol. The alcohol is distilled off and the soap is dissolved in 1 liter of alcohol of 56° Tralles. The solution is titrated against a solution of calcium chlorid of known strength and is so standardized that 1 cc. = 0.0005 gram of CaO.

This solution can be utilized in determining calcium salts in sugar products, provided that the titration be conducted only with neutral or alkaline solutions; that for each titration a few drops of strong ammonia be added; that not more than 20 cc. soap solution be employed, for fear of obscuring the end reaction; for titration of masse cuite, second and third products, their solutions in water are heated to about 40° C.; the lather formed upon completion of titration should be about 1 cm. thick and should not disappear for ten minutes. The method ordinarily employed in determining caustic lime by titrating, adding 90 per cent alcohol and again titrating (after filtration) is pronounced to be very inaccurate.

Oester.-ungar. Ztschr. Zuckerind., 1892; Ztschr. Ver. Rübenzucker-Ind., **42**, 69; Chem. Centralbl., 1892, i, 650; Jour. Chem. Soc., **62,** 1377. B. B. R.

XI.—SPECIAL METHODS FOR MASSE CUITE.

258. BRETON, July, 1892.—*Analysis of masse cuite* (*Pertes á la cuite*).

On the account of the lack of perfect homogeneity in masse cuites, and or the difficulty of securing a representative sample when the small weights ordinarily employed are taken, the author takes a large sample (about 2 kilograms), dissolves it in distilled water, and makes the solution of approximately the same density as the original sirup. Aliquot parts of this can be used for the various determinations and the relative proportions of the different constituents can be readily compared with those of the sirup itself.

Bull. Assoc. Chim. France, **10,** 126. B. B. R.

259. SIDERSKY. D., November, 1892.—*Analysis of sugar products* (*Note relative á l'analyse des produits de sucrerie*).

In determining solids in masse cuite and other products (especially low grade products) by means of density, great differences are noted in the results, as compared with the figures secured by direct estimations. This is due to the fact that the nonsugars, especially the mineral matters, possess densities differing widely from that of sugar.

In arriving at the approximate proportion of solids, the following empirical formula has been employed: Apparent per cent of solids — per cent ash × 0.6 = total solids. This gives an approximation sufficiently close for ordinary work.

Jour. fabr. sucr. **33,** No. 41. B. B. R.

260. HARONIER, J. A.—*Determination of dry matter in masse cuite.*

The author refers to the chief methods in use for determining the amount of dry matter in masse cuite, and to the inexactness of the results secured thereby.

The following process is recommended as giving absolutely accurate results, though it is somewhat time-consuming. A given weight of masse cuite is dissolved in a known weight of water in a flask of 1000 cc. capacity. A known weight of this solution is now allowed to flow, drop by drop, into a bulb, well filled with asbestos, and the bulb and contents are dried until a constant weight is secured. The weight of masse cuite contained in a given weight of the solution being known the percentage of dry matter can be readily calculated.

Bull. Assoc. Chim. France, **11,** 11. B. B. R.

261. VERVINS, E.—*Determination of amount of crystallized sugar in masse cuite.* (*Determination du sucre crystallisé dans la masse cuite en grains.*)

The author proposes to base the determination of the amount of crystallized sugar in masse cuite upon determinations of alkalinity in the masse cuite and in the sirup separated therefrom by means of an apparatus of special construction, which is described in detail. The results of a number of determinations by the processes of Dupont, Sidersky, and the author are given, the method proposed being chiefly advantageous on account of the ease and rapidity of its execution. The formula proposed for use in the calculations is as follows:

$$100 - \frac{100 \times \text{alkalinity of masse cuite}}{\begin{array}{c}\text{alkalinity of sirup separated}\\ \text{from the masse cuite.}\end{array}} = \text{amount of crystallized sugar.}$$

Bull. Assoc. Chim. Belge., **6,** 72. B. B. R.

262. SIDERSKY, D.— *The determination of the amount of crystallized sugar in masse cuite* (*Le procédé Vervins pour la détermination du sucre crystallisé dans la masse cuite en grains*).

Commenting upon the process proposed by Vervins for the estimation of the crystallized sugar in the masse cuite by means of a determination of the alkalinity of the masse cuite and that of the sirupy liquor obtained therefrom, the objection is raised to its employment on the ground of the very considerable errors it may give rise to. Alkalinity determinations frequently can not be made with great accuracy, and a comparatively slight variation in the alkalinity results will give rise to considerable differences in the final results by the employment of the formula given by Vervins.

Sucr. indig., **40,** 483. B. B. R.

263. POCHWALSKY.—*Determinations of the amount of crystals in masse cuite* (*Détermination de la quantité de cristaux dans la masse cuite*).

The details and mode of operation of an apparatus for the separation of the crystals from the sirup are given. The apparatus is provided with connections with an air pump or aspirator in order to facilitate the separation by means of a partial vacuum.

Dodatek, 1892, No. 4; Bull. Assoc. Chim. France, **10,** 376. B. B. R.

264. KARCZ.—*Determination of crystallized sugar in raw sugars and masse cuite* (*Bestimmung des krystallisierten Zuckers im Rohzucker und in Nachprodukten*).

The author recommends the employment of glycerol to effect the separation of sirup from the crystallized sugar in masse cuite, etc. A portion of the glycerol solution, after filtration, is weighed up, clarified with subacetate of lead, and polarized. The amount of sucrose found in the glycerol solution, subtracted from the total sugar present in the material, gives the amount of crystallized sugar.

Oester.-ungar. Ztschrzurkerind, 1894, 21; Bull. Assoc. Chim. France, **11,** 591; Chem. Centralbl., 1894, 1845, 1846. B. B. R.

XII.—SUGARS IN MOLASSES.

265. SIDERSKY D., August, 1892.—*The dextrorotary substances in molasses* (*Les matières dextrogyres contenues dans les mélasses*).

The author refers to differences observed in the determination of sucrose in beet molasses by direct polarization and by Clerget's process, the results by the latter method being almost invariably lower than by the former, whereas if invert sugar is present the reverse should be the case. This difference in the results is generally ascribed to the presence of dextrorotary bodies other than sucrose, and many determinations are made by the method of Creydt, whose formula provides for the estimation of sucrose in the presence of raffinose. The author believes, however, from the result of experiments, that raffinose is frequently absent when its presence may be indicated by this process. For instance, by the action of heat upon pure sugar, products are obtained, which by the application of Creydt's formula show a considerable proportion of raffinose.

The author concludes that the differences referred to are due to—

(1) The incorrectness of the original formula of Clerget.

(2) The products resulting from the action of heat upon sugar.

(3) The excessive addition of lead acetate, consuming a proportion of the hydrochloric acid for the precipitation of the lead and liberating acetic acid which possesses a much lower invertive power.

(4) Lack of care in conducting inversion tests.

In many cases, the author believes the presence of dextrorotary bodies to be imaginary.

Alc. sucr., **1,** 25. B. B. R.

266. PRINSEN-GEERLIGS, H. C., May, 1893.—*The sugars contained in molasses.*

As the result of a large number of tests, the author concludes that the kinds of sugars occurring in molasses are sucrose, dextrose, and levulose; it is highly probable that no others are present, or, if so, only in traces. The dextrose and levulose present do not occur (necessarily) in the proportions in which they are found in invert sugar, but are present in such varying relative quantities that no fixed factor can be established by means of which the glucose content will enable the proportion of saccharose to be determined by direct polarization. The author considers the Clerget method as an accurate means of determining the amount of sucrose under such conditions.

Sugar Cane, **25**, 252. B. B. R.

XIII.—ESTIMATION OF SUCROSE IN MOLASSES.

267. BRUN, E., July, 1892.—*Analysis of molasses* (*Analyse des mélasses*).

The author calls attention to the differences in results of determinations of sucrose in molasses by the methods in common use, and calls attention to the necessity of greater uniformity in such determinations. A comparison is given of the figures secured by the employment of the ordinary method, the method of Clerget (using Sidersky's tables) and that of Creydt, the first named giving the highest results, the formula of Creydt the next highest, while the figures by Clerget's process were the lowest.

Sucr. indig., **40**, 4. B. B. R.

268. SIDERSKY.—*Molasses analysis by inversion* (*Bemerkungen zur Melassenanalyse durch Inversion*).

To 16.19 grams of molasses in 100 cc., not more than 4 or 5 cc. of lead acetate of 30° Baumé should be employed, an addition of bone black before the lead solution being also advantageous. The flask should be heated for fifteen minutes in a water bath previously brought to 70° C. In many cases, 5 per cent of HCl are sufficient for complete inversion.

Sucr. indig., **41**, 379; Chem. Ztg. Rep., **17**, 123. B. B. R.

XIV.—SUCROSE—BORIC ACID METHOD.

269. LACHAUX, February, 1893.—*The employment of boric acid in determination of sugar in scums* (*Emploi de l'acide borique pour le dosage du sucre dans les écumes*).

The employment of boric acid as a substitute for acetic acid in the analysis of scums is very advantageous on account of the readiness with which it effects the decomposition of any sucrate of lime present, nor does it form a soluble salt capable of interfering with the polarization. A large number of comparative tests of this with other methods for the determination of sugar in scums has shown the boric acid process to be highly satisfactory.

Bull. Assoc. Chim. France, **10**, 570.

270. WEISBERG, J.—*The determination of sugar in scums* (*Emploi de l'acide borique pour dosage du sucre dans les écumes*).

In referring to Lachaux's recommendation of the use of boric acid in the analysis of scums, the author states that the objections of the former to the employment of acetic acid are not well founded. On account of the very low invertive power of acetic acid, and the rapidity and completeness with which it decomposes sucrates of lime, its employment has always given satisfaction. Boric acid, on the other hand, forms a bulky, insoluble precipitate of calcium borate, whose presence interferes with the accuracy of the results. The boric acid also combines with the excess of lead, forming a precipitate of lead borate.

Bull. Assoc. Chim. France, **10**, 653. B. B. R.

XV.—FRUIT SIRUPS.

271. DELTOUR, E.—*The analysis of fruit sirups* (*Note sur les sirops alimentaires de pommes, poires, betteraves, etc.*).

In determining reducing sugars, the solution of the sirup, of about 10 per cent strength, is decolorized with lead subacetate and the excess of lead is removed by means of carbonate of soda. The liquid, after being mixed with Fehling's solution, is heated on a boiling water bath for half an hour, and the precipitated cuprous oxid is then washed, ignited, and weighed.

For direct polarization, a solution of about the same strength as the above is used, the excess of lead being precipitated by means of sulphurous acid; for inversion tests 50 cc. of the filtrate are treated with 5 cc. hydrochloric acid and heated for one-half hour on the water bath at 70° C. The various organic acids and pectic principles present are precipitated successively by means of neutral lead acetate, basic acetate, and ammoniacal subacetate solutions.

Each precipitate, after washing and drying, is weighed, and by igniting and weighing again the approximate proportions of the precipitated organic matters can be ascertained by difference.

Bull. Assoc. Chim. Belge., **6,** 57. B. B. R.

XVI.—HONEY.

272. FISCHER, SARTORI, RUNSCHKE, and BUTLER.—*The examination of honey* (*Mittheilungen aus dem chemischen Untersuchungsamte der Stadt Breslau*).

Reference is made to the method of O. Haenle, whereby by means of dialysis it is claimed that addition of starch sirup can be detected with certainty, since by this process a solution is finally obtained which gives a righthanded rotation in case starch sirup is present. The authors employed a dialysis vessel of glass and added some salicylic acid to the honey to prevent fermentation. Results are obtained corresponding to those of E. Dieterich, but they do not show the method of Haenle, however, to be devoid of practical utility, since Haenle dialyzed under somewhat different conditions. An artificial honey, however, by this method appeared from the indications to be a pure honey, while a pure honey after dialysis for two hundred and four hours, showed a slight right-handed polarization.

Jahresber. Chem. Untersuch. Breslau, 1891-'92; Chem. Ztg. Rep., **16,** 349.

B. B. R.

273. BIESTERFELD.—*Detection of adulteration in honey* (*Analyse de miels*).

The author states that an admixture of artificial honey can not be detected with any certainty. Polariscopic investigation of the honey before and after inversion reveals whether cane sugar or dextrose has been added. The mean normal rotatory index of honey is — 5°; quantitative deductions may be gained upon examination of a 20 per cent solution by allowing 3.5° rotation to the right for every 10 per cent of cane sugar added, and a rotation of 5.5° to the right for every 10 per cent of starch sugar. A few honeys have a different normal, notably honey from Coniferæ.

Bulletin de l'Assoc. Belges.; Rev. internat. falsificat., **7,** 42-44.

B.—OTHER CARBOHYDRATES.

1. *Isomaltose.*

274. BAU, A.—*Determination of isomaltose.*

Chem. Ztg., **17,** 499; Chem. Centralbl., 1893, i, 960.

2. *Dextrin.*

275. WILSON, J. A., 1892.—*Action of mercuric cyanid on dextrin.*

The method proposed by Wiley for the determination of dextrin in the presence of dextrose and maltose, mercuric cyanid being used to destroy the latter, has been reviewed. Separate treatment of the substances named showed, when the action was continued for two minutes, that the rotatory power of dextrose was destroyed, that of maltose much reduced, but that of dextrin also perceptibly diminished.

Chem. News, **65,** 169; Agric. Science, **7,** 185; Jour. Chem. Soc., **62,** 1032. W. E. S.

3. *Starch—products of action of diastase.*

276. SCHIFFER, A.—*Noncrystallizable products of action of diastase on starch.*

By the action of diastase upon starch, the author endeavored to obtain substances corresponding to the malto-dextrins of Brown and Morris and of Herzfeld. The only products obtained were dextrin, isomaltose, and maltose, the so-called malto-dextrins being considered merely as mixtures of dextrin and isomaltose.

Inaug. Diss., Kiel, 1892; Neue Ztschr. Rübenzucker, **29**, 167.

4. *Starch—fixation of iodin.*

277. ROUVIER, G.—*Fixation of iodin by starch.*

Author showed earlier (Bull. Soc. Chim. [3], 7, 272) that iodin entered into combination with starch in several proportions. He now finds from a series of analyses that in the presence of an excess of starch the compound formed has the composition $(C_6H_{10}O_5)I_8$.

Compt. rend., **114**, 749, 1366; Bull. Soc. Chim. [3], **7**, 678; Centralbl. Agrik. Chem., **21**, 855, 856. W. F.

5. *Estimation of starch.*

278. GUICHARD, 1892.—*Determination of starch* (*Dosage de l'amidon et action des acides étendus sur la cellulose*).

Invert with dilute HNO_3 (10 per cent) and polarize the solution; in the presence of cellular tissue, starch must first be removed by boiling with oxalic acid and then inverted by nitric acid.

Jour. pharm. chim. [5], **25**, 394; Bull. Soc. Chim. [3], **7**, 554–560, 630; Agric. Science, **7**, 186. W. E. S.

279. BAUDRY, A., 1892.—*Determination of starch.*

A weighed quantity of starch (2.688 grams for Laurent's polariscope) is boiled with 80 to 90 cc. of water and 0.4 to 0.5 grams of salicylic acid for forty to fifty minutes, made up to 200 cc. with ammonia, filtered and polarized in a 400 mm. tube. The reading of circular degrees corresponds to the percentage. The method is applicable either to commercial starch or to potatoes or similar substances.

Chem. Centralbl., 1892, i, 339, 509; Agric. Science, **7**, 186. W. E. S.

280. DELTOUR.—*Baudry's method for estimation of starch* (*Stärkebestimmung nach Baudry*).

Similar to foregoing in substance.

Bull. Assoc. Chim. Belge., **6**, 154; Chem. Ztg. Rep., **17**, 42.

281. GIRARD, AIMÉ, and FLEURENT.—*Apparatus for approximate determination of starch in potatoes.*

A simple apparatus for catching and measuring water displaced by a given quantity of potatoes.

Jour. d'agric. prat., 1892, i, 374–376; Centralbl. Agrik. Chem., **21**, 858, 859. W. F.

6. *Carbohydrates of special plants: Agavose.*

282. MICHAUD, G., and TRISTAN, J. F.—*Researches on the sugar of Agave americana.*

Description of separation and properties of agavose, a saccharose, optically inactive, but yielding levorotatory inversion products; strongly reducing Fehling's solution, yielding no mucic acid on oxidation, and crystallizing readily.

Amer. Chem. Jour., **14**, 548–550.

7. *Carbohydrates of coffee.*

283. EWELL, ERVIN E.—*The carbohydrates of the coffee berry.*

By extraction with alcohol, precipitation by basic lead acetate, inversion and treatment with Fehling's solution, 6.25 per cent of sucrose was found, but little reducing sugar. The water insoluble residue yielded 4.2 to 4.6 per cent of furfuraldehyde upon distillation with HCl, and to 5 per cent NaOH solution it yielded a gum containing galactose and pentose groups ("galacto-xylan"). Little or no mannose was found.

Amer. Chem. Jour., **14,** 473–476.

8. *Pentosans—properties.*

284. STONE, W. E., and TEST, W. H., 1893.—*Notes on xylose.*

A record of the authors' observations on the multirotation of xylose studied by Tollens and Parcus is given.

Amer. Chem. Jour., **15** [3], 195–197. J. W. F.

285. TOLLENS and SCHULZE, 1892.—*Analytical properties of pentoses.*

The specific rotation of xylose is found to vary somewhat with temperature and rotation; for a 10 per cent solution and 20° C., $(A)_d$=18.794. Neither arabinose nor xylose is readily changed by sulphuric acid. After twenty hours' boiling with 4 per cent acid only slight decomposition had occurred, and after thirty-two hours, 84 per cent of arabinose and 73 per cent of xylose were unchanged.

Landw. Vers.-Stat., **40,** 379–384; Agric. Science, **7,** 180. W. E. S.

9. *Pentosans—Estimation.*

286. TOLLENS, GÜNTHER, and DE CHALMOT.—*The determination of pentosans.*

The method of distillation with HCl and precipitation and weighing of the furfurol by phenylhydrazin is found satisfactory. By this method xylose yielded 52 per cent and arabinose 43 per cent of furfurol; while wheat straw gave 21.90 per cent; corn cobs, 30.94 per cent, and brewers' grains, 27 per cent.

Landw. Vers.-Stat., **40,** 11–17; Agric. Science, **7,** 178, 179. W. E. S.

287. TOLLENS, GÜNTHER, and DE CHALMOT.—*A source of error in determining pentosans.*

It is found that glucuronic acid and its derivatives yield furfurol in considerable quantities, as well as do pentosans. This would be a serious source of error in determining pentosans as furfurolhydrazin were not glucuronic acid so rarely found.

Ber. chem. Ges., **25,** 2569; Agric. Science, **7,** 179. W. E. S.

288. FLINT, E. R.—*Gravimetric determination of the pentaglucoses.*

The author describes his modification (Ber. chem. Ges., **25,** 2912) of the method of de Chalmot and Tollens, and gives results on various woods and gums.

Jour. anal. Chem., **7,** 190. A. L. W.

289. FLINT, E. R., and TOLLENS, B.—*Determination of pentaglucoses* (*Ueber die Bestimmung von Pentosanen und Pentosen in Vegetabilien durch Distillation mit Salzsäure und gewichtsanalytische Bestimmung des entstandenen Furfurols*).

The authors maintain that the gravimetric method for the estimation of furfurolhydrazone, first proposed by de Chalmot and Tollens, is more reliable than Günther's titration method. The latter gives high results, for the reason that other substances (levulinic acid, etc.) formed during the heating with hydrochloric acid are contained in the distillate and combine with a portion of the phenylhydrazin. The presence of these impurities does not affect the accuracy of the gravimetric method, since they are not precipitated in dilute solutions by phenylhydrazin. The presence of starch and cane sugar introduce an error for which no remedy has been found. Cellulose does not interfere. The authors recommend the following modifications of the method of de Chalmot and Tollens: (1) The quantity of sodium chlorid in the 500 cc. of neutralized distillate should be always 81.5 grams, which is the quantity obtained by neutralizing 400 cc. of 12 per cent hydrochloric acid. To secure this amount 10.2 grams sodium chlorid are added for each 50 cc. which the distillate falls short of 400 cc. After neutralizing, phenylhydrazin acetate is added and the solution made up to 500 cc. (2) The separation of the precipitate is aided by stirring for one-half hour with a mechanical stirrer. (3) The hydrazone is collected in a filtering tube, glass wool being substituted for asbestos. (4) The air drawn through the filtering tube during the drying of the precipitate is first passed through sulphuric acid, to remove moisture, and through a tube filled with calcium carbonate to remove any sulphuric acid which may be carried over mechanically.

The formulæ for calculation of results are as follows:

I. $\text{Arabinose} = \text{Hydrazone} \times 1.229 + .0177$.
II. $\text{Xylose} = \text{Hydrazone} \times 1.031 + .001$.
III. $\text{Pentose} = \text{Hydrazone} \times 1.13 + .0083$.
IV. $\text{Furfurol} = \text{Hydrazone} \times .516 + .0252$.

Ber. chem. Ges., **25**, 2912, **27**, R, 143; Landw. Vers.-Stat., **42**, 381–407; Jour. Chem. Soc., **64**, ii, 52. A. L. W.

290. DE CHALMOT, G., 1893.—*Soluble pentoses in plants.*

The author has investigated the soluble pentoses and hexoses in the leaves and colorless bark of a large variety of plants, and has devised the following method for the estimation of small quantities of furfuraldehyde. When anilin, furfuraldehyde, and an alcoholic solution of acetic acid are mixed, a very intense red color immediately appears, which is used as the basis of a comparative color test. One cubic centimeter of a weak solution of furfuraldehyde in 5 per cent acetic acid was treated in a long tube with 1 cc. of a 1 per cent solution of anilin in .95 per cent alcohol. Another tube, containing 1 part of furfuraldehyde in 10,000 was prepared in the same way, and in comparing the stronger solution was diluted with a mixture of equal parts of the alcoholic anilin solution and 5 per cent acetic acid. The following formulæ; where H = value sought and α = number cubic centimeters diluent used, were used in the determination:

If the solution of unknown strength be the stronger,

$$H = \frac{\alpha + 2}{2} \times \frac{1}{10,000}$$

If the standard solution be the stronger,

$$H = \frac{2}{\alpha + 2} \times \frac{1}{10,000}$$

Am. Chem. Jour., **15**, 1, 21–38. J. W. F.

291. DE CHALMOT, G., 1893.—*Pentosans in plants.*

The author has continued his work on the pentosans, with special reference to the influence of germination upon pease and corn. He uses the following methods for the determination of—

(1) *Total pentosans.*—Distil slowly, adding 10 cc. hydrochloric acid every twenty minutes until 100 to 110 cc. are distilled; neutralize with soda, and bring to 200 cc. with 19.3 per cent sodium chlorid solution, and precipitate the furfuraldehyde after the directions of Flint.

(2) *Pentosans insoluble in dilute acids.*—Ten grams of the substance are heated with 200 cc. hydrochloric acid of 1.25 per cent; filter, dry, and distil with 100 cc. of 12 per cent hydrochloric acid.

(3) *Soluble pentosans.*—Determine the total amount of carbohydrates and the amount of furfuraldehyde obtained by distillation with hydrochloric acid, as described.

Amer. Chem. Jour., **15** [4], 276–285, **16**, 218–228; Jour. Am. Chem. Soc., **15**, 618; Exp. Sta. Record, **5**, 1103, 1104; Ber. chem. Ges., **26**, R, 791; **27**, R., 422. J. W. F.

292. KRUG, W. H.—*Some modifications of the method of de Chalmot and Tollens for determination of furfurolhydrazone.—An improved method for the gravimetric estimation of furfurolhydrazone.*

The author reviews the work of the past twenty years on pentoses. The improvements on the method of de Chalmot and Tollens proposed are as follows: (1) After adding phenylhydrazin, the liquid is allowed to stand over night to insure complete precipitation. (2) To obviate the inconvenience occasioned by the adhering of the precipitate to the beaker, the latter is not cleaned, but simply rinsed with water, which is afterwards poured through the filtering tube. After all salt is removed, both the beaker and the tube are dried at a gentle heat, the precipitate is dissolved in 30 to 40 cc. of alcohol, and the solution evaporated in a small flask at 60° C., forcing a current of air through the flask during the drying.

Jour. anal. Chem., **7,** 68; Jour. Chem. Soc., **64,** A, 556; Chem. Centralbl., 1893, i, 861. A. L. W.

293. HOTTER, E., 1893.—*A new method for the quantitative determination of pentoses in plants (Eine neue Methode zur quantitativen Bestimmung der in den Vegetabilien vorkommenden Pentosanen).*

The author uses pyrogallol instead of phenylhydrazin for precipitating the furfurol formed, and proceeds as follows: 5 to 10 grams of material are distilled with hydrochloric acid (1.06 sp. gr.) until the distillate fails to show a reaction for furfurol with anilin acetate. The distillate is diluted to a definite volume (400 or 500 cc.) with the same or somewhat stronger hydrochloric acid, and an aliquot of 20 to 30 cc. is heated with an excess of pyrogallol (0.5 to 1 gram) in a closed tube at 100° to 110° C. for from one to two hours. The precipitate thus formed is brought on a tared filter, washed, dried at 103° C., and weighed. The weight of the precipitate divided by 1.974 gives the amount of furfurol in the aliquot, from which the percentage of pentoses is found by the ordinary calculations. The presence of a large excess of pyrogallol is said to do no harm, and under the provisions prescribed the reaction between it and the furfurol is said to be quantitative. It is important that a sufficient quantity of condensed furfurol solution be taken, and that the furfurol solution contain at least 12 per cent of hydrochloric acid. The author suggests carrying on the distillation with hydrochloric acid under diminished pressure to prevent the formation of humic substances.

Chem. Ztg., **17,** 1743–1745; Exp. Sta. Record, **5,** 613; Chem. Centralbl., 1894, i, 115, 116; Jour. Soc. Chem. Ind., **13,** 424. J. P. S.

294. COUNCLER, C., 1894.—*A new method for the quantitative determination of furfurol (Ein neues Verfahren zur quantitativen Bestimmung von Furfurol, bezw. den in den Vegetabilien enthaltenen Pentosanen).*

Five grams of substance are distilled with 12 per cent hydrochloric acid until the distillate fails to show a furfurol reaction. The distillate is diluted with 12 per cent hydrochloric acid to 250 to 500 cc., care being taken to make the solution homogeneous. Bring 20 to 50 cc. of the solution into a flask of proper size with not too great an excess of phloroglucin; stopper tightly with a glass stopper, shake several times and allow it to stand about twelve hours. Then bring the resulting precipitate on a weighed filter, dried at 98°, wash, dry at 98° and weigh. By dividing the weight of the precipitate by the proper factor the amount of furfurol in the flask is obtained, and from this the amount of furfurol contained in the total distillate. From this by using the Tollens factor the amount of pentosans contained in the substance may be determined.

Chem. Ztg., **18,** 966–968.

10. Cellulose—Composition of wood.

295. LINDSEY, J. B., February-April, 1892.—*The composition of wood (an extended study bearing upon cellulose determination).*

In conclusion the author remarks: "The investigation into the composition of wood, and into the cellular structure of the plant, show no groups of very complex substances. Amyloid, soluble in water, and yielding various sugars by hydrolysis, has been recognized as a cellular constituent of many seeds. The hemicelluloses are very largely distributed in our fodder plants and grains. These two groups of substances, together with starch, the sugars that are already formed in the plant, resins, and coloring matters make up the nitrogen-free extract."

Cellulose proves itself not always to be a polymeric anhydrid of dextrose, but rather a combination of anhydrids, yielding different sugars upon inversion. Lignin is closely bound with cellulose, and little is known of its chemical nature.

It is quite probable that our methods for separating the different groups of substances are faulty, in that these substances undergo changes in composition during the treatment. Thus hemicelluloses, when extracted with dilute alkali, change into soluble matters, behaving like acids. Schulze's reagent probably works changes in the cellulose. The constitution of the cellulose molecule is not yet fully known. Until it is, the determination of groups of kindred substances rather than of individual materials must be adhered to.

Agric. Science, **7**, 49–57, 97–106, 153–176. W. F.

296. THÖRNER, W., March, 1893.—*Determination of woody fiber by means of the centrifugal process (Genaue und schnelle Bestimmung der Holzfaser in Futtermitteln mit Hülfe der Centrifuge).*

In the determination of fiber by this process, a tube with an enlargement just below its mouth is employed, while a stirring rod, flattened and broadened at the end, is used to aid in agitation of the contents.

One gram of the finely divided sample is used, and if a considerable amount of fat is present it is subjected to treatment with 20 cc. of ether, several times in succession, the tube being rotated in the centrifugal and the ether decanted. The ether is now evaporated off in the water bath, 30 cc. hot water added, and the whole heated in a boiling-water bath for ten minutes, with constant stirring.

Ten cubic centimeters of dilute sulphuric acid (50 cc. concentrated acid in 1 liter) are now poured in and the tube heated thirty minutes longer, stirring the contents constantly. After removal of the glass rod, the tube is rotated in the centrifugal machine at the rate of 2,000 revolutions per minute for three to four minutes. The liquid, which is somewhat turbid, is now poured through a dried and weighed filter of about 35 cc. capacity, most of the insoluble matter being left in a compact mass in the bottom of the tube. The tube, after about 40 cc. of hot water has been poured in, is heated in the water bath for ten to fifteen minutes, with constant stirring; the tube is now rotated as before, the liquid poured upon the same filter, and this operation of washing again repeated. To the residue in the tube is next added 30 cc. hot water and 10 cc. of caustic potash solution (5 grams KHO in 1 liter) and the heating for one-half hour is conducted as before. The operations of rotating in the centrifugal and washing by decantation with water are executed as before described, the fiber brought upon the filter, washed with alcohol and ether, dried and weighed in the usual manner. Eight determinations can be carried on at once and can be completed in from three to four hours. Results of analysis of a number of feed stuffs by this process are reported.

Chem. Ztg., **17**, 394; Analyst, **18**, 138; Exp. Sta. Record, **4**, 767. B. B. R.

297. GABRIEL, S.—*Estimation of cellulose.*

Hönig's method (Chem. Ztg., 1890, 53) by treating with glycerol at 210° gives too high results, the residue being rich in ash and nitrogen. The Weende method was found to give better results, and a solution of potassium hydroxid in glycerol heated to 150° gave fair results.

Ztschr. physiol Chem., **16,** 370–386; Jour. Chem. Soc., **62,** 923; Analyst, **17,** 155; Vjschr. Chem. Nahr., **7,** 186. J. W. F.

298. HANAUSEK, T. F., 1894.—*Solvent for cellulose* (*Lösungsmittel der Cellulose*).

The author has examined the new solvent for cellulose, suggested by Cross and Bevan, which consists of a solution of 1 part of zinc chlorid in 2 parts of anhydrous acetic acid (glacial acetic acid), and has found that while it is an excellent clarifier and dissociator, it is not properly a solvent of cellulose.

Chem. Ztg., **18,** 441. J. P. S.

C.—PROTEIDS.

299. OSBORNE, T. B., 1892.—*Crystallized vegetable proteids.*

The author has investigated the properties of the crystallized vegetable proteids of the Brazil nut, hemp seed, the castor bean, flaxseed, the oat kernel, and the squash seed, and finds—

(1) That the globulins of the Brazil nut and oat kernel are distinct substances. Their composition is as follows:

	Brazil nut.	Oat kernel.
Carbon	52.18	52.18
Hydrogen	6.92	7.05
Nitrogen	18.30	17.99
Sulphur	1.06	.53
Oxygen	21.54	22.34
	100	100

They also differ in many of their qualitative reactions.

(2) That the crystalline globulins of the hemp seed, castor bean, squash seed, and flaxseed are almost identical in composition, as follows:

	Hemp seed.	Castor bean.	Squash seed.	Flaxseed.
Carbon	51.28	51.31	51.66	51.48
Hydrogen	6.84	6.97	6.89	6.94
Nitrogen	18.84	18.75	18.51	18.60
Sulphur	.87	.76	.88	.81
Oxygen	22.17	22.21	22.06	22.17
	100	100	100	100

This in addition to the similarity of their deportment toward reagents.

Amer. Chem. Jour., **14,** 8, 662–689. J. W. F.

300. OSBORNE, T. B.—*Proteids in flaxseed.*

Author is unable to state exactly the factor for calculating the proteids in flaxseed from the nitrogen found, but from his studies is led to suggest 5.55 instead of 6.25.

Conn. State Sta. Report, 1892, 138–142; Exp. Sta. Record, **4,** 11; Amer. Chem. Jour., **14,** 629–661. E. W. A.

301. MILLIAU, ERNEST, May 29, 1893.—*The methods of testing oils and fats.*

A general discussion of methods for determination of the purity of commercial fats and oils.

Jour. Am. Chem. Soc., **15** (1893), 153–172.

302. Lane, Nathaniel L., June, 1893.—*Muter's method for the analysis of fat and oil—a simplification.*

Author states that he had gotten very satisfactory results by the following simplified modification of Muter's process (Analyst, 1889, April). Precipitate the lead soaps of the saponified fat in a 600 cc. Erlenmeyer flask; cool under running water, giving flask a circular motion, by means of which the lead soaps are made to adhere to the sides of the flask; when perfectly cool, wash by decantation with hot water, rinse once with alcohol, add 120 cc. redistilled ether, attach to reflex condenser and boil in water bath till the oleate is completely dissolved and the insoluble soaps are finely powdery. Then cool thoroughly. This boiling in ether diminishes greatly the time required for filtration. The remainder of the process is just the same as the original by Muter.

Jour. Am. Chem. Soc. **15,** 110–113. W. F.

2. Chlorophyll extract.

303. Étard, A.—*Proximate analysis of chlorophyll extract.*

Author proposes to extract the dried, coarsely ground plant, first with CS_2: (1) The solvent is driven off at low temperature and the residue extracted with cold alcohol until it comes off colorless; the insoluble greenish residue, crystallized from benzol, and then acetic ether (in presence of animal char) white, often beautiful crystal, either solid hydrocarbons, alcohols, glycols, or higher glycerins. (2) The alcohol extract is distilled *in vacuo,* taken up with 2 per cent KOH, shaken with ether, which dissolves alkaloids, alcohols, glycols and chlorophyll substances. (3) The KOH solution is then acidified and again agitated with ether, which dissolves the various saturated and unsaturated fatty acids; the original plant residue from CS_2 extraction is then extracted with warm alcohol, and latter removed by distillation *in vacuo.* (4) The residue is mixed with one or more volumes of pulverized glass, and shaken with cold alcohol; the insoluble residue is rubbed with ether, which leaves undissolved extractives free from chlorophyll, of brownish green tint, and astringent taste. (5) The ether dissolves chlorophyll substances. (6) The alcohol extract from the CS_2 insoluble matter is distilled *in vacuo*; the residue gives up much chlorophyll to ether, indeed most of it is found here. (7) The residue from (6) insoluble in ether always contains colorless, nitrogenous extractives.

Compt. rend., **114,** 1116; Centralbl. Agrik. Chem., **22,** 401–403. W. F.

3. Estimation of pure fat.

304. Gebek, L.—*A method for determining the pure fat in fodder* (*Ueber Fettextraction*).

The author finds that ether extract, purified by gypsum or bone black, is pure fat, but results are not concordant. "Spanish earth," such as is used for the purification of wines, is a more satisfactory purifying agent. Before using, it should be made into a paste with water, treated with sulphuric acid to remove carbonate evaporated, ignited, and finally ground, so that it passes a 2 mm. mesh. Five grams of air-dry fodder are mixed with an equal volume of "Spanish earth," placed in an extraction tube over a layer of 3 to 4 cm. of "Spanish earth," and extracted for four to five hours with anhydrous ether. If fodders are dried at 100° C. previous to the extraction, the results may be much too low.

Ztschr. angew. Chem., 1893, 254. Landw. Vers.-Stat., **43,** 193. Chem. Centralbl. 1893, i, 1093; 1894, i, 442. Exp. Sta. Record, **4,** No. 10; **5,** 801. Chem. Ztg., **18** Rep. 15. Jour. Soc. Chem. Ind., **12,** 713. Ber. chem. Ges., **26,** R, 556.
A. L. W.

4. Detection of adulteration of vegetable oils.

305. Gassend, A.—*Detection of sesame oil in olive oil.*

Some specimens of African olive oil give a red coloration with the sugar and hydrochloric acid test, similar to that obtained with sesame oil. Spectroscopically, they are easily distinguished; chemically, they may be distinguished by adding a little of a 10 per cent solution of sodium hydrogen sulphite, the red coloration produced by olive oil being discharged very quickly, whereas that produced by sesame oil is not discharged for twelve to forty minutes.

Rev. Internat. Sci. Fals. Aliment, **5,** 98; Chem. Centralbl., 1892, i, 459; Jour. Chem. Soc., **62,** 1133. J. W. F.

306. Villavecchia, V. and Fabris, G.—*The use of furfurol for the detection of sesame oil in oil mixtures.*

Ztschr. angew. Chem., **17,** 505; Chem. Ztg., **17,** Rep. 236; Ber. chem. Ges., **26,** R, 829; Jour. Soc. Chem. Ind., **13,** 69.

307. Anonymous.—*Detection of adulteration of cocoanut and palm oils.*

The addition of oils less soluble in absolute alcohol may be readily detected by the insolubility of the mixed oils in that medium; 5 cc. of cocoanut oil, previously washed with 95° alcohol, should dissolve in 10 cc. absolute alcohol at 30° to 31° C.; and 5 cc. of palm oil, treated in like manner, in 20 cc. of absolute alcohol.

Jour. pharm. chim.; Rev. internat. falsificat., **6,** 82, 83. W. F.

308. Gantter, F.—*Detection and approximate estimation of cotton-seed oil in lard and olive oils.*

The author discards Becchi's silver nitrate test as nonconclusive. The iodin absorptions of lard and of cotton oil, 23 to 27 per cent and 43 to 45 per cent, respectively, differ widely enough to allow of the calculation, within 10 per cent, of the proportion of cotton oil present. To detect smaller amounts than 10 per cent, 1 cc. of the dry, melted fat or oil is dissolved in 10 cc. of light petroleum in a test tube; a single drop of sulphuric acid is added, and the whole well shaken. Pure lard gives only a straw-yellow to feeble reddish-yellow colored liquid, from which, on repose, dark, reddish-yellow drops slowly settle out, leaving a nearly or perfectly colorless upper liquor. Olive oil acquires a rather darker color at first, but behaves like lard on standing. In presence of cotton oil, a deep brown to black color is immediately formed which remains unchanged on standing even for a long time.

Ztschr. anal. Chem., **32,** 303–308; Jour. Chem. Soc., **64,** A, 440. J. W. F.

5. *Estimation of cholesterin.*

309. Obermüller, K.—*Determination of cholesterin.*

Present methods are erroneous because of solubility of alkaline soaps in ethereal solution of cholesterin, or to the solubility of cholesterin in the solutions of the soaps. To obviate this error, dissolve the fat in ether, add concentrated sodium ethylate (for 1 gram of fat, 0.15 grams of sodium, and 1.5 cc. alcohol); let stand at ordinary temperature with frequent agitation; at the end of three hours the ethylate will have disappeared, and saponification is complete; filter, wash with dry ether, unite the ethereal liquid, evaporate to dryness at 100° to 120° C.; take up residue in a few cc. absolute ether, filter, evaporate, again dry at 120° C., and weigh. It is possible after saponification with the ethylate to take up the product in CS_2, add standard solution of bromin in CS_2 just to persistent coloration, and, assuming that the bromin had been used to form cholesterin dibromid, to compute the quantity of the former.

Bull. Soc. Chim. [3] **7,** 658, 659. W. F.

310. Lewkowitsch, J.—*Determination of cholesterin.*

Boil in flask attached to inverted condenser, with 1.5 part by weight of glacial acetic acid, filter, wash with cold water till washings are neutral. Then boil the filter and contents with an excess of standard alcoholic KOH, assigning the formula $C_{26}H_{43}O,.C_2H_3O$ to the solid; cholesterin may be computed from the amount of acetic acid liberated to form potassium acetate.

Or the cholesterin may be dissolved in 50 cc. chloroform, 25 cc. of an alcohol solution of iodin and $HgCl_2$ (Hübl) are added; a blank is made at the same time. The iodin entering into combination is determined by titration with hyposulphite, 68.3 of iodin corresponding to 100 of cholesterin.

Ber. chem. Ges., **25,** 65; Bull. Soc. Chim., [3] **8,** 671–865. W. F.

311. SCHULZE, G., and FRANKFURT, L.—*Concerning the lecithin content of some vegetable substances.*

Landw. Vers.-Stat., **43,** 307; Chem. Ztg., **18,** Rep., 17.

E.—OTHER PLANT CONSTITUENTS.

1. *Essence of mustard.*

312. SCHLECHT, A.—*Determination of essence of mustard.*

A method is proposed dependent upon the digestion of the material (cabbage, colza, etc.) with a large excess of water acidulated with a little tartaric acid to secure the decomposition of potassium myronate; subsequent distillation into alkaline permanganate of potassium, reduction of manganese by alcohol, oxidation of any sulphurous acid thus formed by action of iodin dissolved in potassium iodid, estimation of sulphuric acid as usual, and the multiplication of the figures obtained for $BaSO_4$ by 0.425 to give the allyl sulphocyanid.

Landw. Vers.-Stat. (1892), **41;** Rev. internat. falsificat, **6,** 136, 137. W. F.

2. *Tannin.*

313. FLEURY, G.—*Estimation of tannin.*

Method depends upon the absorption of the tannin by pulverized white of egg, and consequently the increased dry weights of the latter. It is applicable to the tannins of wine, of red rose leaves, but those of gallnuts are very slowly and incompletely absorbed.

Jour. pharm. chim., [5] **25,** 499; Bull. Soc. Chim., [3] **7,** 687. W. F.

314. KOCH, R.—*A serious source of error in the gravimetric estimation of tannin.*

Results vary largely with the hide used as an absorbent and with the coloring matter present in the substance analyzed. Minute directions are given for the selection and preparation of the hide used.

Dingl. polyt. Jour., **280,** 141–159; Bull. Soc. Chim., [3] **8,** 1043, 1044. W. F.

3. *Test for polyatomic phenols.*

315. STAHL, J.—*Colored reactions of molybdic acid with various polyatomic phenols.*

The author finds that the orange-colored reaction observed by Hager between ammonium molybdate and the tannins occurs also in the case of the polyatomic phenols having two hydroxyls in ortho positions; other phenols are not sensibly affected. This reaction occurs also in the case of the bromin, nitro, and ethereal derivatives; if the phenol is itself a colored body its color is strongly exaggerated. An aqueous solution of molybdic acid, ordinarily giving a blue color with organic bodies, in this case gives the yellow or brown tint. The author attributes it to the active reduction of molybdic oxid MoO_3 to the brown MoO_2. Sodium tungstate is affected similarly.

Ber. chem. Ges., **25,** 1600; Bull. Soc. Chim., [3] **8,** 1035. W. F.

4. *Guanidins.*

316. PRELINGER, O.—*Picric acid as a general reagent for the guanidins.*

Brieger observed that methyl guanidin is entirely precipitated by picric acid; Emich noted that it was equally efficient in isolating guanidin; the author extends this observation to a triphenyl guanidin and to phenyl guanidin, the reaction being very delicate.

Monatsh. f. Chem., **13,** 97–100; Bull. Soc. Chim., [3] **8,** 1039, 1040. W. F.

317. SCHULZE, E.—*Nessler reagent as a test for guanidin.*

The author states that the white precipitate is readily obtained in very weak solution, is sharply distinct from the ammonium precipitate, and that by other reactions the white guanin precipitate is readily distinguished.

Ber. chem. Ges., **25,** 661; Bull. Soc. Chim., [3] **8,** 1038. W. F.

5. Alkaloids, etc.

318. HULSEBOSCH, L.—*Determination of alkaloids in narcotic and other extracts* (*Bestimmung von Alkaloïden in narkotischen und anderen Extracten*).

Pharm. Centralbl., 1893, **8,** 101; Ber. chem. Ges., **26,** Ref. 339.

319. ALLEN, A. H.—*Volumetric determination of alkaloids.*

Chem. News, **66,** 259; Ber. chem. Ges., **26,** Ref. 389.

320. BARTHE, L.—*A volumetric method for alkaloids.*

Dependent upon their indifference to phenolphthalein, but their property of turning litmus blue.

Compt. rend., **115,** 512; Ber. chem. Ges., **25,** 871.

321. GRANDVAL, ALEX., and LAJOUX, HENRI.—*Determination of thein in plants* (*Dosage de la caffein dans les vegetaux*).

Wet the powdered substance with ammoniacal ether (1 ammonia, 5 ether), shaken together just before adding, stir thoroughly, and transfer to continuous extraction apparatus. Extract completely with 50 cc. of chloroform; drive off the solvent completely; flow the dry residue with 1 cc. of official sulphuric acid; let stand some moments; leach repeatedly with hot water, a little at a time; filter each portion through a wet filter, unplaited, keeping the funnel covered; add excess of ammonia to filtrate; evaporate to dryness on water bath, to get rid of coloring matters, which become insoluble in chloroform; take up with this solvent, filter solution, evaporate slowly on water bath; a pure white, crystalline thein, free from tannin, is secured.

In the case of roasted coffee the coloring matters persist; therefore, the acid aqueous filtrate from the first chloroform residue is made alkaline, and agitated in a separatory funnel with successive portions of chloroform (2 or 3); evaporate the solution obtained, and satisfactory crystals will be yielded.

Rev. internat. falsificat., **6,** 168–170. W. F.

METHODS FOR SPECIAL FOOD MATERIALS.

A.—SEEDS.

322. VON ASBÓTH, A., 1894.—*The analysis of seeds and grains* (*Die Analyse der Samenarten*).

The author had previously proposed a method for the analysis of tuberous plants, and now applies it to the analysis of vetches and similar plants. The water, ash, and fat are determined in the usual ways. The fat is extracted from several portions of the vetch meal with ether, and these portions are dried in air until free of ether, and are then used in the estimation of the other constituents. Five grams of the extracted meal are weighed off and mixed with several cubic centimeters of 0.10 potassium hydrate and about 50 cc. of cold water. The alkali is added because the wet meal has an acid reaction, and would hinder the perfect solution of the proteids present. No more alkali should be added than just enough to neutralize the solution. In mixing the meal with water the odor of prussic acid was noticed, which, according to Ritthausen, arises from the amygdalin present. He was unable, however, to isolate the amygdalin successfully. After settling, the clear fluid is decanted off, and the residue washed again with cold water. This is continued until the meal settles out quickly, which results after four or five decantations. Now bring the meal on a tared filter, and wash it well with water. The remaining substance is washed once with alcohol and ether, and then dried, first at 40°, then at 110°. The filtrate is added to the decanted liquid and slightly acidified with acetic acid. The legumin which is separated is collected on a tared filter, washed with water acidified with acetic acid, and dried, first in a vacuum over sulphuric acid and finally at 110°. By steaming the filtrate a flocculent precipitate may appear. This is albumen or some legumin remaining in solution, and is collected on a tared filter and treated as the legumin. After weighing the precipitate incinerate it and deduct the ash from the total weight. In the remaining liquid the rest of the nitrogen may be determined by the Kjeldahl method, modified by Asbóth. From this nitrogen the asparagin is calculated.

The substance insoluble in water is divided into three parts—one for starch, one for nitrogen, and one for ash. The nitrogen is determined as legumin (factor, 16.87). The cellulose is determined by difference.

The carbohydrates in the extracted meal are determined by the baryta method. Because of the acid reaction of the wet meal it is treated by the baryta method, as usual, and the acid titred direct. The resulting base is deducted. By deducting the starch from the total carbohydrates the dextrin is obtained. He was unable to determine the sugar in the meal.

Since asparagin contains 21.97 per cent of nitrogen, the protein must not be reckoned from the total nitrogen, but the legumin and asparagin determined separately.

Chem. Ztg., **18**, 32, 33. J. P. S.

323. SCHULZE, E., 1894.—*The analysis of the seeds of plants* (*Ueber die Analyse der Pflanzensamen*).

Chem. Ztg., **18**, 799–802. J. P. S.

B.—CATTLE FOODS IN GENERAL.

324. EMMERLING, A.—*Concerning a simple method for the detection and approximate determination of sand in fodders, meals, etc.*

Ztschr. anal. Chem., **33**, 46; Chem. Ztg., **18**, Repert., 30; Chem. Centralbl., 1894, i, 486; Ber. chem. Ges., **27**, Ref. 209.

325. WOLL, F. W., 1894.—*Recent progress in the analysis of cattle foods*

Jour. Am. Chem. Soc., **16**, 174–178; Exp. Sta. Record, **5**, 937. J. P. S.

326. LEANDRE.—*Practical methods for analysis of flour* (*Méthodes pratiques d'analyses des farines*).

The author recommends, for determination of moisture, drying 5 grams to constant weight in an oil bath at 100° to 110° C., weighing at half-hour intervals. In determining gluten, the caution is given to allow the mixed dough to stand two hours under a watch glass before washing to permit "complete hydration." For determination of ash, 5 grams are slowly ignited in a muffle or in a platinum dish. The sodalime, and Kjeldahl methods and Bolierre's ammonimeter are recommended for nitrogen determination. Aqueous extract is found by converting 100 grams of the flour to a paste, diluting to a liter with cold water, filtering, rejecting first part, and evaporating to dryness 50 cc. of the filtrate. To determine fat, 5 grams is successively percolated with 6 to 10 portions of ether, percolate evaporated, dried at 100° C. for an hour, and weighed.

Rev. internat. falsificat., **6**, 133–136, and 153–156. W. F.

327. SMITH, JOHN BARKER.—*New method of valuation for bread, flour, albumens, etc.* (*2d part*).

Bull. Acad. Roy. de Belgique [3], **22**, 528; Ber. chem. Ges., **25**, 603.

C.—SUGAR IN TOMATOES.

328. CALDWELL, G. C.—*Determination of sugar in the tomato.*

Neither the polariscope or Fehling test considered wholly reliable for direct determination of sugar in tomatoes. In the method used tomatoes were dried at 100° C., the ground residue extracted with boiling water or 90 per cent alcohol in extraction apparatus. Extraction with the latter solvent and determination of sugar by gravimetric copper method gave very satisfactory results.

N. Y. Cornell Sta. Bull., No. 49, 301, 302; Exp. Sta. Record, **4**, 802. E. W. A.

D.—OIL CAKE—MUSTARD OIL CONTENT.

329. ULBRICHT.—*The mustard oil content of rape and oil cakes (Ueber die Menge ätherischen Senföls welche Rapskuchen liefern).*

Schuster and Mecke (Chem. Ztg., 1892, p. 1954) found that the heating which rape seed was submitted to previous to extraction of its oil developed a substance which yielded mustard oil when the press cake was mixed with water. This generation of mustard oil when cake was mixed with water, had previously been taken as indicating the presence of mustard seed in the cake. Ulbricht controlled the above experiments and arrived at the same conclusion, namely, that the presence of mustard oil did not necessarily indicate adulteration of the oil cake, but was a natural product. Method of determining mustard oil in feeding stuff given.

Landw. Vereinssch. Balt. Cent. Ver., 1893, 65, 66; Exp. Sta. Record, **4**, No. 11.
E. W. A.

330. SCHUSTER and MECKE.—*The mustard oil product from rape and oil cakes.*

Chem. Ztg., 1892, 1954; Exp. Sta. Record, **4**, No. 11.

E.—OIL CAKES—MOISTURE AND OIL.

331. AITKEN, A. P., 1894.—*A rapid and accurate method of determining the moisture and oil in linseed cakes and other feeding stuffs.*

Jour. Am. Chem. Soc., **16**, 114–122; Chem. Centralbl., 1894, **1**, 743–745.

332. LEATHER, J. W.—*Detection of castor-oil seed and croton seed in feeding stuffs.*
Analyst, 1892, 121–127; Exp. Sta. Record, **4**, 211.

333. HILTNER, L.—*Simple method for recognizing adulterations of peanut cake.*

Landw. Vers.-Stat., **40**, 351-355; Exp. Sta. Record, **4**, 381.

F.—TEA.

334. DVORKOVITCH.—*Determination of caffein, tannin, and fermentation products of tannin in tea (Les analyses de différentes sortes de thé chinois).*

In discussing the bases for the valuation of tea, the author proposes the following method for analysis of tea. Thoroughly extract the powdered tea with hot water, treating three times with 200 cc. of boiling water and twice boiling with 200 cc. of water, using 10 grams of tea. Unite the extract and make up to a liter. Treat a part with petroleum ether, agitating thoroughly and treating three times. To 600 cc. thus treated add 600 cc. $Ba(OH)_2$, 4 per cent solution; shake and filter at once. To the filtrate add 100 cc. of common salt solution (20 per cent), and shake three times with chloroform, using altogether about 400 cc. of this solvent. Decant, rinse, evaporate from tared dish. The use of lime or magnesia for boiling with the tea lead to too low results.

In determining tannin the Löwenthal principle is used, attention being called to the fact that the rate of oxidation is slower, with more rapid addition of permanganate. Use 40 cc. of tea extract above mentioned; add 500 cc. water, 25 cc. indigo carmine (25 cc.=20 cc. permanganate), and 25 cc. sulphuric acid (1:5); add 23 cc.

permanganate (130 cc.=100 cc. $\frac{N}{10}$ oxalic acid); and then add the reagent, 2 or 3 drops per second, until reaction is complete; use no more than 38 cc. of the permanganate, taking less tea extract, if need be. (In standardizing the indigo, add 11 cc. permanganate, then 2 or 3 drops per second, and finally only 1 drop per second.)

In determining decomposition product, to 80 cc. of the tea extract add 20 cc. 4 per cent $Ba(OH)_2$, filter, and make up 50 cc. filtrate as in determining tannin. Add 18 cc. permanganate, then 2 or 3 drops per second. The amount of permanganate increases with the degree of fermentation.

Rev. internat. falsificat., **6**, 65-68, 78-81. W. F.

335. CAZENEUVE and BIÉTRIX.—*Determination and extraction of the caffein of tea (Dosage et extraction de la caféine du thé).*

Authors give an outline of the methods of Pelletier and Caventou, of the French Codex, of Loche, of Paul and Cownley, and of Cazeneuve and Caillot. They find that by the methods of Loche and of the Codex 3 per cent is extracted, by that of Paul and Cownley 3.80, and by that of Cazeneuve and Caillot 3.73. The last two methods give a colorless product, the others a colored one. The method of Cazeneuve and Caillot is preferred on account of its rapidity.

Rev. internat. falsificat., **6**, 13, 14. W. F.

336. HANAUSEK, EDOUARD.—*Detection of steeped tea leaves (The épiusé ou déjà employe et son analyse).*

The author has studied the indices of refraction of successive hot aqueous extracts of tea, and concludes that the refractive indices diminish with the degree of exhaustion. The refractive index is the numeral expression of the total water-soluble matters of the decoction and not of the tannin alone; nor do they show any relationship to the color of the tea (black, yellow, or green). Teas made up of one-half weight of steeped tea leaves have a considerably lower refractive index than whole teas of the same kind, so that the index may be used as a means of detecting such fraud in large measure.

Rev. internat. falsificat., **5**, 210, 211, **6**, 209, 210. F.

G.—COCOA.

337. SUSS, P.—*Determination of theobromin in cocoa.*

Pulverize with an equal part of sand; extract 6 grams of the mixture for six hours with petroleum ether; the residue, thus deprived of fat, is mixed with 200 cc. of water and 6 grams of pure lead oxid, and then heated with stirring for thirty minutes; filter; treat residue a second time with 100 cc. of water. Reduce united aqueous extracts to 10 cc., mix with 100 cc. of chloroform, shake three minutes, separate, distill off most of the chloroform, and transfer residue to a tared vessel, carefully washing the distillation flask with a little chloroform, and evaporate slowly on water bath.

Ztschr. anal. Chem., 1893, 32-57; Rev. internat. falsificat., **6**, 175. W. F.

338. SCHROEDER, M., 1892.—*Estimation of sugar in cocoa preparations.*

The estimation of grape sugar in mixed cocoas or chocolates can not be effected by Fehling's solution on account of the small amount of invert sugar in commercial cane sugar. By using the polariscope good results may be obtained, since, if grape sugar is present, the rotation after inversion will be less than it should be. Fermentation may be used as a check, the amylin, which is invariably present in grape sugar being then easily recognizable.

Ztschr. angew. Chem., 1892, 173-183; Jour. Chem. Soc., **64**, ii, 100. J. W. F.

H.—COFFEE SUBSTITUTES.

339. MOSCHELES and R. STELZNER.—*Analysis of coffee substitutes.*

Imitation coffee yields a higher aqueous extract than the real article. Twenty-five grams of the compound is rubbed in a mortar, introduced into a liter flask, and digested with 500 cc. of water on the water bath for half an hour. The mixture is made up to 1 liter. Fifty cubic centimeters of the extract is evaporated and weighed. Pure coffee gives 20 to 30 per cent of extract, while some coffee substitutes yield as high as 72 per cent.

Chem. Ztg., **16,** 281, 282; Jour. Chem. Soc., **62,** 1534, 1535 J. W. F.

I.—VINEGAR.

340. IMHOFF.—*To detect the adulteration of vinegar by mineral acids.*

Stir several drops of a dilute solution of methylanilin violet with a drop of the vinegar on a porcelain plate; if mineral acids are present, the color will quickly change, becoming green if HCl or H_2SO_4 be present, blue if carbolic acid is present.

Jour. d. l. Soc. de Pharm. d'Anvers; Rev. internat. falsificat., **6,** 49. W. F.

341. THUMANN, A.—*To detect sulphuric acid in vinegar.*

Put small pieces of white paper in the vinegar, contained in a porcelain dish, and slowly evaporate at a moderately low temperature. If sulphuric acid be present, the paper will be blackened.

Rev. internat. falsificat., **6,** 128. W. F.

ALCOHOLIC LIQUORS.

ABSOLUTE ALCOHOL.

342. SQUIBB, EDWARD R., 1893.—*Absolute alcohol (second paper.)*

Author, believing that by use of cold-lime process and simple distillation more perfect freedom of pure alcohol from water might be effected than had hitherto been attained, experimented upon the subject. As an indication of the degree of "absoluteness" obtained, the specific gravity was used. The author describes some very accurate apparatus used for this determination, for which see original.

The author did not, however, succeed in passing his earlier figure, 0.79350 sp. gr., nor even in fairly equaling it. He finds the sodium process inferior to the cold-lime process.

Jour. Am. Chem. Soc., **15,** 126–140. W. F.

GENERAL METHODS.

343. GOSSART, EMILE.—*A new process for the analysis of alcohol and alcoholic liquors (Nouveau procédé d'analyses des alcools).*

The method proposed is one of comparison, depending upon the fact that a drop of pure alcohol will roll upon a surface of like nature, while it will disappear in a pure liquid of unlike nature, and that in the presence of a mixture drops will behave as if in the presence of their likes. The vapor tension by which the drops are sustained is, however, modified by degree of dilution, as well as by temperature and the slope of the surface, increase of dilution tending to diminish the degree of rolling and to cause the disappearance of the drop. An increase of viscosity, on the other hand, increases the delicacy of the process. It is claimed that 0.1 per cent of each impurity can be certainly detected and estimated by this process, and that without great expenditure of time or labor.

Rep. Pharm., July, 1892, 301; Rev. internat. falsificat., **6,** 26–29. W. F.

344. GRÜNHUT, L.—*A difficulty in Röse's process for the estimation of alcohol.*

The oxidation of the alcohol to carbonic anhydrid and water is found to depend to a great extent on the amount of sulphuric acid. If too much acid is added, an independent decomposition of the permanganate will set in, rendering the results untrustworthy. The process can not, therefore, be recommended.

Chem. Ztg., **15,** 847, 848; Jour. Chem. Soc., **62,** 1031. J. W. F.

345. LALIEU, A.—*Determination of dry extract and glycerol in alcoholic liquors, vinegar, etc.* (*Le dosage de l'extrait sec dans les essais chimico-pharmaceutiques.*)

Use only 1 gram of substance (in the case of wines, set the weight at 0.995, as 5 milligrams will be lost in the one and one-half minutes consumed in weighing). Drop a little more than 1 cc. in a tared watch glass, take up excess by a strip of filter paper, dry for thirty minutes in a water bath, cool on a piece of metal, and weigh. (In case of wines and vinegar, add 0.002 grams for glycerol volatilized, but not in case of beers.) In wines rich in extract, beers, etc., dilute to two or four volumes with water, weigh out 1 gram, and proceed as above. Glycerol may be determined in red wines (but not in white or sweet wines nor in beer) and in vinegar by estimating loss by further drying for two or three hours.

Jour. d. l. Soc. de Pharm. d'Anvers; Rev. internat. falsificat., **6**, 103, 104 and 116–119. W. F.

346. VON BITTO, BELA.—*Reaction for monovalent alcohols* (*Analyse des alcools*).

A monovalent alcohol to which 1 or 2 cc. of a 0.05 per cent aqueous solution of methyl violet and then 0.5 cc. of an alkaline polysulphid are added, and the mixture shaken, takes on a clear coloration, becoming cherry to violet-rose; otherwise the liquid colors bluish-green, and later precipitates violet-red flecks, leaving the liquid yellow. Polyvalent alcohols do not produce the reaction.

Chem. Ztg., **15** (1893), 611; Rev. internat. falsificat., **7**, 61. F.

DISTILLED LIQUORS, ETC.

347. MOHLER, E.—*Examination of commercial spirits* (*Gang zur Untersuchung von Handels-Sprit*).

Aldehyde is determined by the author colorimetrically by means of rosanilin bisulphite, the tint being compared with that obtained by dissolving ethyl aldehyde in a 50 per cent solution of pure alcohol.

Furfurol is also determined colorimetrically by means of anilin along with acetic acid, 0.1 milligram of furfurol being capable of detection in this manner. The alcoholic homologues are estimated by the colorimetric method by means of sulphuric acid after removal of aldehyde, furfurol, and bases by the use of anilin phosphate. The nitrogenous constituents are determined very much in the same manner as in the process generally employed for water analysis, distilling first in presence of sodium carbonate and afterwards with alkaline permanganate. The sample under examination must, however, first be heated along with a little phosphoric acid in order to expel alcohol. The free acid (calculated as acetic) is determined by titration with $\frac{N}{10}$ potash solution, using phenolphthalein as indicator. Extract is estimated by drying for seven hours in a Gay-Lussac oven the residue obtained by the evaporation of 50 cc. of the sample.

Ann. chim. phys., **23** [6], 121; Ztschr. anal. Chem., **31**, 583. B. B. R.

348. BARDY, C.—*Approximate method for determining higher alcohols in ethyl alcohol.*

The method is intended to apply to "head and queue" alcohol, and the distillery residues known as "essential oils." It consists in the separation of butyl and amyl alcohol by the solvent action of CS_2 in the presence of saturated salt solution; the subsequent separation of the CS_2 by evaporation in the presence of strong H_2SO_4, and the formation of the acetic esters by the addition of glacial acetic acid. The combined esters are separated by saturated salt solution, measured at 15° C., and multiplied by coefficient 0.8 to get the percentage of combined butyl and amyl alcohol present in the original liquid. Propionic acid is separated with the ethyl alcohol from the saline liquid residue from the washing with CS_2, by distillation until distillate stands at 50 alcoholometric degrees. Its percentage may then be approximately estimated by the method of Barbet or of Gossart.

Compt. rend., May 23, 1892; Rev. internat. falsificat., **6**, 119–121; Bull. Soc. Chim. [3], **7**, 685. W. F.

WINE.

A.—GENERAL.

349. BOTTLER, M.—*Wine analysis* (*Weinuntersuchung*).

Ztschr. Nahr. Untersuch., 1892, 97; Ztschr. angew. Chem., 1892, 470.

B.—EXTRACT, SUGAR AND ALCOHOL.

350. WETZKE, T., June, 1893.—*Alcohol and extract determinations in rich wines* (*Alkohol- und Extractbestimmung in gehaltreichen Weinen*).

Reference is made to the determination of alcohol by the direct distillation process and also by difference. In this latter method the specific gravity of both the original sample and also of the dealcoholized wine is taken, the liquid being in this latter case made up to its original volume. The difference between the specific gravities is subtracted from 1,000, and from the figure thus secured the alcohol percentage can be obtained by reference to a table. Alcohol determinations in a number of wines by both processes are reported, the regular distillation method yielding considerably higher figures in every case. Results of a series of estimations of extract, both by the direct and the indirect methods, is also given, the figures for the latter process being taken from the tables of both Schultze and Hager. In the indirect method for extract estimation, the dealcoholized wine is diluted to its original volume and its density accurately determined. In the results reported the figures for the indirect method, as taken from Schultze's table, show a very close agreement with those obtained by the direct method.

Chem. Ztg., **17**, 836. B. B. R.

351. JEAN, FERDINAND.—*Note on the determination of dry extract in wines* (*Observation sur le dosage de l'extrait sec dans les vins*).

The author proposes that the determination be made in the presence of a known quantity of litharge, thus avoiding the errors due to volatilization of glycerol, size of vessel, time of drying, etc., and permitting the hastening of the operation by use of a temperature higher than that of the water-bath.

Rev. internat. falsificat., **6**, 119; Chem. Centralbl., 1893, i, 960. W. F.

352. MEDICUS.—*Quick extract estimation in wines* (*Rasche Extractbestimmung im Wein*).

Five cubic centimeters of wine are evaporated on a weighed watch glass and the residue dried for half an hour in a water oven. In ordinary wines the results agree moderately well with those secured by the official method. Sweet wines should first be diluted to ten times their volume, or so much that the amount of extract should not exceed 0.2 gram. The figures thus secured are about 0.5 per cent higher than those obtained by the indirect method.

Ber. Ver. Bay. angew. Chem., 1893, 80; Ztschr. anal. Chem., **32**, 362. B. B. R.

353. NESSLER, J., June, 1893.—*Determination of extract and sugar in new wines* (*Bestimmung des Extract- und Zuckergehaltes in jungen Weinen*).

Allusion is made to the changes in composition taking place in new wines, especially in those which are slightly sweet, and these changes, occurring during the after fermentation, cause a considerable diminution in the proportion of sugar and extract. The results of determination of sugar and extract in eleven samples of wine are given, duplicate determinations being made with an interval of three days between the tests. The results of these experiments indicate the necessity of making the extract and sugar estimations simultaneously, since a quite considerable discrepancy in the figures secured is exhibited.

Chem. Ztg., **17**, 878. B. B. R.

354. MULLER, J. A.—*Estimation of the dry residue from wine.*

Rates of desiccation for wines containing various quantities of glycerol are deduced, thus permitting the absolute residue at 100° to be calculated from the residue after twelve hours desiccation, which is a quantity depending on the percentage of glycerol present, and the quantity of wine taken for evaporation, provided that the residue is not much more than 0.1 gram, and that the desiccation is carried on exactly in accord with the prescribed method.

Bull. Soc. Chim. [3], **9,** 6–10; Jour. Chem. Soc., **64,** A, 501. J. W. F.

355. BORNTRÄGER, ARTHUR, April 15, 1894.—*Is it needful to remove the lead from wine clarified by lead acetate? (Ist es nötig für die Zuckertitrierung nach Fehling die mit Bleiessig ausgefällten Weine von Blei zu befreien?)*

The author states that the presence of lead may be ignored in wines containing 1 per cent or more of sugar. If less than that amount of sugar is present, however, he finds that the lead ferrocyanid which is formed interferes with the end reaction.

Ztschr. angew. Chem., 1894, 232. W. D. B.

356. BORNTRÄGER, ARTHUR.—*Estimation of alcohol in wine (Nochmals zur Alkoholbestimmung im Weine durch Destillation und Ermittelung der Dichte des Destillates.)*

The author insists on the correctness of his former statement, that it is unnecessary to neutralize ordinary wines before distillation in the determination of alcohol. Even when the amount of acetic acid present is as high as 1.12 per cent, the error in the alcohol determination amounts to only about 0.3 per cent. To prevent foaming or bumping, the use of sand or pumice stone is suggested, in place of tannin, as directed by Borgmann.

Ztschr. angew. Chem., 1894, 108–111. W. D. B.

C.—RECOGNITION OF POTATO SUGAR.

357. FRESENIUS, W.—*Recognition of potato-starch sugar in wines.*

The statement of Neubauer that the substances precipitable from potato-starch sugar were not fermentable, having been based on experiments made with beer yeast, and the investigations of v. Raumer having shown that the behavior of these substances was not identical with different kinds of yeast, the following experiments were made: A specimen of dextro-rotating wine, examined by Medicus (Ztschr. anal. Chem., **30,** 665–688), and an optically inactive wine were evaporated to sirups and mixed, the former with a 10 per cent solution of completely fermentable invert sugar, and the latter with a 10 per cent solution of potato-starch sugar containing the so-called "unfermentable substances." Under the influence of pressed yeast at 30° C. both lost their rotating power completely in four days. In a similarly conducted experiment with a nonpolarizing wine mixed with potato-starch sugar and beer yeast the solution still showed a rotation of 5° after thirteen days. It was noticed that the wines were very apt to become moldy on the surface, and after the growth of mold the dextro-rotatory constituents were found to have disappeared. The same was the case with nitrates.

Ztschr. anal. Chem., **30,** 669–672; Jour. Chem. Soc., **62,** 922. J. W. F.

358. MEDICUS, L., and IMMERHEISER, C.—*The fermentability of dextrin.*

Some specimens of wine which the authors had condemned as adulterated with potato-starch sugar were by other analysts pronounced genuine on account of the complete fermentability of their dextrorotatory constituents. The authors confirm the fact of the complete fermentation, but found it to take place very slowly. Specimens giving originally a dextrorotation of 0.45° to 0.83° lost this entirely in from twenty-five to twenty-eight days after mixing with pressed yeast. The substances so fermented were proved to be dextrins by their behavior toward Fehling's solution after inversion, a much greater increase in the reducing power being found, after treatment capable of saccharifying the dextrin, than after simple inversion of the cane sugar present. These results were confirmed by experiments upon potato-starch sugar. Two hundred and fifty cubic centimeters of a 16 per cent solution, mixed with a little ammonium and potassium phosphates and 2 grams of pressed yeast, fell off in rotary power from 17° to 7° in eleven days at ordinary temperatures, and on further addition of yeast and elevation of temperature to 30° C. the polarization was reduced to about 1° in the course of thirty-four days. In a second experiment on solutions of the same strength without phosphates, but with more yeast, the rotation fell to 0° after thirty-two days. In a third experiment dextrin, precipitated by alcohol from potato-starch sugar and thoroughly washed with alcohol, was employed with a precisely similar result. The conclusion is that dextrin is with difficulty completely fermentable under the influence of vigorous pressed yeast at a favorable temperature.

Ztschr. anal. Chem., **30,** 665–668; Jour. Chem. Soc., **62,** 922. J. W. F.

D.—GLYCEROL ESTIMATION.

359. SUHO, E.—*Glycerol determination* (*Glycerinbestimmung*).

Arch. Hyg., **14,** 305; Ztschr. angew. Chem., 1892, 430.

360. BAUMERT and SCHAUMANN.—*Determination of glycerin in wine* (*Glycerinbestimmung in Wein*).

Evaporate 50 to 100 cc. of the wine to half its volume with 1 to 2 grams pure precipitated calcium carbonate and distill in a current of superheated steam. To the distillate, which should measure about 200 cc., add 10 to 12 grams caustic potash free from oxalic acid. When cool add saturated permanganate solution until the color becomes permanent. Boil for one-half hour and decolorize by passing a stream of sulphurous acid gas into the solution. Add concentrated acetic acid and heat until sulphurous acid is driven off. Precipitate with calcium chlorid solution. When the precipitate of calcium oxalate and sulphate has settled, collect on an asbestos filter and wash with hot water. Boil the precipitate together with the asbestos in dilute sulphuric acid and titrate with permanganate solution which has been standardized with oxalic acid. Ninety parts of oxalic acid correspond to 92 parts of glycerin.

Arch. Pharm., **231,** 324; Ztschr. angew. Chem., 1892, 716; Ber chem. Ges., **25,** Ref., 918; Ztschr. anal. Chem., **31,** 717. A. L. W.

361. SALVATORI, S.—*Oxidation of glycerol in acid solution.*

The details of oxidation with permanganate are given.

Staz. sper. agr. Ital., **21,** 130–140; Jour. Chem. Soc., **64,** ii, 247. J. W. F.

362. SALVATORI, S.—*Estimation of glycerol in wine and other fermented liquids.*

The author gives the details of the application of the permanganate method to wines.

Staz. sper. agr. Ital., **21,** 141–145; Jour. Chem. Soc., **64,** ii, 248. J. W. F.

363. JOHNSTONE, W.—*Determination of glycerol by potassium permanganate.*

Author finds in Wanklyn's process that not only are traces of alcohol converted to oxalic acid by the action of the permanganate, but also any free butyric acid. He finds that the substitution of potassium manganate is preferable in the titration of the oxalic acid.

Jour. Soc. Chem. Ind., **10,** 203; Bull. Soc. Chim. [3], **8,** 1036, 1037. W. F.

364. MANGOLD, C.—*Determination of glycerol by the method of Benedict and Zsigmondy.*

Author states that heating after addition of permanganate is needless. Presence of butyric acid does not influence the result, because permanganate in alkaline medium does not attack it in the cold; prolonged boiling is required to transform it in such medium into oxalic acid. Check tests made include mixtures containing 90 per cent of butyric acid. Recommends solution of 0.1 to 0.4 gram of glycerol in 300 cc. water, addition of 10 grams KOH, and about ten times as much permanganate, in 5 per cent solution, as there is glycerol present, keeping the mixture cool. After thirty minutes add H_2O_2 in slight excess of amount needed for discharge of color; make up to a liter, mix, and filter off 500 cc. through a dry filter; heat for thirty minutes to decompose excess of H_2O_2, cool to 60°, and, after adding sulphuric acid, titrate with standard permanganate.

Ztschr. angew. Chem., 1891, 400; Bull. Soc. Chim. [3], **8**, 1037, 1038. W. F.

365. LECCO, M. F.—*Determination of glycerol in sweet wines* (*Zur Glycerinbestimmung im süssen Weine*).

The author finds that when his method, previously described (Chem. Ztg., **14**, 504; Ber. chem. Ges., **25**, 389c.) is applied to sweet wines the glycerol is contaminated with sugar. This difficulty can be obviated by using more calcium hydrate, proceeding as follows:

Mix 10 cc. of the wine with 1 gram dry-powdered calcium hydrate, add 10 grams quartz sand, and evaporate almost to dryness; extract the residue 4 or 5 times with hot absolute or 96 per cent alcohol and filter into a 100 cc. flask. Evaporate the filtrate. Dissolve the thick sirupy residue in 5 cc. absolute alcohol, add 7.5 cc. (or, better, 10 cc.) of ether, and cork tightly. Let stand some hours until the solution becomes clear. Pour off the liquid, filter if necessary, evaporate, and finally dry one hour in a drying cell and weigh.

Ber. chem. Ges., **25**, 2074, 2075; Chem. Ztg., **16**, 504; Ztschr. anal. Chem., **31**, 469; Rev. internat. falsificat., **6**, 15; Jour. Chem. Soc., **62**, 1263. A. L. W.

366. PROSKAUER, B.—*Determination of glycerol in fermented liquors* (*Ueber Glycerinbestimmung in vergohrenen Getränken*).

The following method, it is claimed, yields satisfactory results: 100 cc. wine are evaporated to about 30 cc., together with a drop of H_2SO_4 and 6 cc. phosphotungstic acid (50 per cent solution). The precipitate is filtered and washed, and the filtrate, with addition of dry calcium hydrate and quartz sand, is evaporated on a water bath. The mass is then rubbed up in the dish and brought into a Soxhlet's apparatus. After thorough extraction with 96 per cent alcohol, the extract obtained is evaporated in the flask to a sirupy consistency. Next add 25 cc. alcohol ether (2:3), close flask, and after thorough shaking let settle clear; the clear liquid is poured into a long-necked flask, and after addition of 10 cc. alcohol ether is evaporated and dried in water oven.

Pharm. Central-H., **13**, 369; Chem. Ztg. Rep., **16**, 228. B. B. R.

E.—TANNIN.

367. CHAIRMONTE, T.—*Estimation of tannin in wine.*

The author gives a record of some experiments upon the subject, and recommends the following process: 50 cc. of wine are evaporated on the water bath to about one-third, made distinctly alkaline with ammonia, and treated with 10 to 15 cc. of zinc acetate solution and 2 cc. of 10 per cent ammonia. The whole is heated for a few minutes on the water bath, allowed to cool, filtered through a plaited filter, and washed with cold water. The zinc tannate is quickly dissolved in 15 cc. dilute sulphuric acid, 20 cc. indigo-carmine solution added, and made up with water to about 400 cc. To this, permanganate solution is gradually added until the color changes from greenish to gold. The solutions were as follows: Permanganate 0.2 per cent, zinc acetate 1 per cent, sulphuric acid 20 per cent.

Staz. sper. agr. Ital., **20**, 337-350; Jour. Chem. Soc., **64**, ii, 311, 312. J. W. F.

F.—ACIDITY, ETC.

368. WENGER, W. H., 1892.—*Solubility of cream of tartar in diluted alcohol.*

The author gives a table of the solubility of cream of tartar in alcohol of various strengths at 25° C.

Am. Chem. Jour., **14**, 8, 624, 625. J. W. F.

369. ACKERMAN, E., July, 1892.—*Estimation of tartar in sweet wines* (*Ueber die Bestimmung des Weinsteins in Süssweinen nach der Methode von Berthelot und Fleurieu*).

Reference is made to an article of Petrowitsch, published some years since, giving the analysis of a wine which showed no cream of tartar according to Berthelot and Fleurieu's method. In experimenting with a sweet Grecian wine a result similar to that observed by Petrowitsch was obtained, no crystalline deposit being visible, but in its stead a brown slimy precipitate. This, which was supposed by Petrowitsch to consist of coloring matter, was found to be chiefly sugar along with a little tartar. The sugar present was supposed to interfere with the crystalline separation of the cream of tartar, and the wine was accordingly fermented; after addition of ether alcohol a marked crystalline deposit was obtained, free from any slimy particles. It is therefore considered necessary to first ferment sweet wines before employing the method of Berthelot and Fleurieu.

Ztschr. anal. Chem., **31**, 405; Rev. internat. falsificat., **7**, 85. B. B. R.

370. ANONYMOUS.—*Detection of free tartaric acid in wine* (*L'acide tartarique en vin*).

Instead of precipitating directly, agitate the extract from 200 grams of wine with alcohol, which dissolves the free acid, but not tartar; evaporate, take up residual extract with water, and precipitate by adding potassium acetate and a few drops of acetic acid; 1,000 parts of tartar=701 parts anhydrous tartaric acid; the purity of the precipitate should be subjected to the ordinary check tests.

Rev. internat. falsificat., **6**, 123. W. F.

371. SOSTEGNI, L.—*Some points in the determination of density in the color of wines and in the determination of free tartaric acid.*

Staz. sper. agr. Ital., **23**, 10; Analyst, **18**, 91. F. W. W.

372. MULLER, J. A.—*The determination of acidity due to fixed and volatile acids in wine* (*Sur la détermination de l'acidité due aux acides fixes et volatils du vin*).

Total acidity is determined by dilute standard barium hydrate, phenolphthalein indicator. Carbonic acid is then removed by action of an exhaust pump, and acidity again determined. Volatile acids are now driven off by heating in a porcelain vessel over a gas or alcohol lamp at a temperature not higher than the hand will bear. The residue must not be brown. A little water is added and driven off in like manner, and this repeated. Solution in water and final titration follow. To secure accurate results the fixed acid must considerably exceed the volatile; if it does not, add tartaric or other fixed acid in known amount and correct later. Instead of direct titration, boiling with known quantity of barium hydrate in excess and back titration with HCl is preferable. Reduced pressure may be dispensed with in driving off the CO_2, if the liquid be brought barely to boiling point, the vapors blown off, and the operation repeated. In direct evaporation of the wine some of the volatile fatty acids are lost by entrainment with the alcohol; some retained in the pasty residue. By comparative determination the author is led to recommend that the figures for volatile acid obtained as above be multiplied by the coefficient 1.167, and that that for the fixed acids by 0.08 times the corrected figure for volatile acids.

Bull Soc. Chim. [3], **7**, 830–832; Ann. chim. phys., **25** [6], 118; Ztschr. anal. Chem., **32**, 109. W. F.

G.—INORGANIC MATTERS.

1. *Sulphurous acid.*

373. RIPPER, M.—*Determination of sulphurous acid in wine* (*Die schweflige Säure im Weine und deren Bestimmung*).

The sulphurous acid in sulphured wines exists partly as the free acid or alkali sulphite and partly as aldehyd-sulphurous acid. The total sulphurous acid may be determined by treating successively with normal potassium hydrate and sulphuric acid (1:3) and titrating direct with $\frac{N}{50}$ iodin solution; the free acid by essentially the same process, except that no potassium hydrate is added. Aldehyd-sulphurous acid is obtained by difference. When the gravimetric method is used barium sulphate is best purified by bromin and hydrochloric acid.

Jour. prakt. Chem., **46**, 428; Ber. chem. Ges., **26**, Ref. 199. A. L. W.

374. KLEIBER, A.—*Determination of sulphurous acid in wine* (*Zur Bestimmung der schwefligen Säure im Weine*).

The following process is proposed, by means of which it is claimed that one can readily determine whether or not wine samples exceed the allowed limit of 80 milligrams per liter, and it is also possible to combine the preliminary tests with the final quantitative determination. One hundred cubic centimeters of wine are distilled in the usual way in a stream of carbon dioxid. In the receiver are placed 2.5 cc. $\frac{N}{10}$ iodin solution and about 25 cc. water and a few drops of starch solution are also added. When the distillation has proceeded for about twenty minutes without decolorization taking place, the sample contains less than 80 milligrams sulphurous acid per liter, and a quantitative determination can be avoided. In case the solution is decolorized, 2.5 cc. more of iodin solution are added, the liquid being titraed with $\frac{N}{10}$ sodium thiosulphate solution.

Schw. Woch. Chem. Pharm., **31**, 45; Chem. Ztg. Rep., **17**, 54. B. B. R.

2. Phosphoric acid.

375. MORGENSTERN and PAWLINOW.—*Determination of phosphoric acid in wines.*

Uranium method gives too high results Ashing unnecessary. Two hundred cubic centimeters of the wine are boiled a short time until most of the alcohol has evaporated; 20 cc. nitric acid (sp. gr. 1.38) are added and the boiling continued until nitrous fumes are no longer given off. The solution is nearly neutralized and phosphoric acid is determined by the citrate method.

Jour. Russ. Chem. Soc. (1891), **24** [1], 341; Ber. chem. Ges., **25**, Ref. 920; Chem. Ztg. Rep., **16**, 228; Vjschr. Chem. Nahr., **7**, 317, 318; Chem. Centralbl., 1893, i, 503; Jour. Chem. Soc., **64**, A, 389. A. L. W.

3. Chlorin.

376. SEIFERT, W.—*Estimation of chlorin in wine* (*Zur Bestimmung des Chlors in Wein*).

Ztschr. anal. Chem., **31**, 186; Ber. chem. Ges., **25**, Ref., 598; Ztschr. angew. Chem., 1892, 441.

4. Adulteration.

377. QUANTIN, H.—*Detection of wine treated with barium salts for the removal of calcium sulphate.*

Directions are given for determination of excess of chlorin, tartaric, citric, phosphoric and nitric acids, as well as of barium.

Compt. rend., **114**, 369; Centralbl. Agrik., Chem., **21**, 694, 695. W. F.

378. DE COLLIS, H.—*Detection of alum in wines.*

Two methods are given: (1) 100 cc. of wine are evaporated to dryness in a porcelain dish on a sand bath, and carefully treated until no more products of decomposition are evolved. The carbonized residue is pulverized in the dish, covered with dilute acetic acid, allowed to remain for twenty-four hours, boiled, filtered, evaporated to dryness, and heated until there is no further odor of acetic acid; water is then added, the mass again boiled, filtered, and ammonium chlorid and ammonia added. Any aluminum artificially introduced into the wine (as sulphate) is now precipitated, the aluminum tartrate naturally occurring in wine having been rendered insoluble by the incineration. (2) A definite quantity of the wine is evaporated to dryness in a platinum dish, and completely calcined, the process being hastened by the addition of ammonium nitrate. The ash is taken up with hot hydrochloric acid, filtered, the residue washed free from acid and sulphates, and the sulphates in the solution estimated. By comparison with the sulphates in the original wine, the amount of aluminum sulphate added (and decomposed by ignition) is ascertained.

L'Orosi, **15,** 118–120; Jour. Chem. Soc., **62,** 1523. J. W. F.

379. Cavalli, Anselme.—*Detection of vegetable coloring matters in wine* (*Recherche de quelques matières colorantes végétales dans les vins*).

Author uses a 5 per cent soap solution for this purpose. (A soap containing 66.5 per cent fatty acids, 7.3 per cent alkali, and 26.5 per cent of water gave good results.) To 2 cc. of the wine add 10 cc. of the soap liquor. Pure wine will give a coloration, obscure red or rose, or pale reddish violet; wine colored with mauve, an intense violet color; with campeachy, a brown or coffee-black, deepening; with fernambuc, a brick red, which, unlike that of pure wine, becomes yellow on boiling; with phytolacca, a bright red, permanent; with cochineal, an obscure red, growing paler; with indigo, a beautiful blue. Observe tints at intervals, make check tests on pure wine, and see that "goudron" colors are absent.

Rev. internat. falsificat., **7,** 62. F.

380. Maronby.—*Detection of adulteration in wines.*

The appearance of spots on various test papers is used for this purpose.

Bull. Soc. Chim. [3], **9,** 13–16; Jour. Chem. Soc., **64,** A, 501. J. W. F.

Beers.

A.—Alcohol.

381. Harvey, S.—*Notes on Tabarie's process for the indirect estimation of alcohol in beers.*

The distillation process was found to yield an alcohol of lower density than by Tabarie's method (calculated alcohol = 1 — sp. gr. of extract — sp. gr. of beer); author shows this to be due to the carbonic acid in the beer, and states that fairly satisfactory results may be obtained by a complete or nearly complete expulsion of the carbonic acid in the beer.

Analyst, **17,** 1–4. F. W. W.

382. Allen, A. H.—*Note on Tabarie's method of determining alcohol.*

Author gives some experiments with artificial solutions, which show that both Tabarie's formula $\frac{L}{E} = S$, and Blunt's formula $S = 1000 - (E-L)$ give erroneous results. (L = sp. gr. of liquid, E = sp. gr. of extract, S = sp. gr. of spirit.)

Analyst, **17,** 5. F. W. W.

383. Tornoë, H., February 20, 1894.—*Estimation of alcohol in beer by means of the ebullioscope* (*Étude sur l'ébullioscope et son emploi pour le dosage de l'alcool dans les bières*).

The author finds that it is possible to make an accurate determination of alcohol in beer in 20 minutes with the aid of the ebullioscope and densimeter.

Bull. Soc. Chim., 1894 [3], **11,** 116–120. W. D. B.

B.—Fermentable substances in wort.

384. Bau, A., October, 1892.—*The determination of fermentable substance in beer worts* (*Ueber die Bestimmung der vergährbaren Substanz in Bierwürzen*).

Experiments are reported in which a large variety of yeast cultures were employed, the fermentable substance in a beer wort, as shown by the use of the different varieties of yeast, being accurately determined. The beer worts were previously carefully sterilized, and the determinations were conducted in sterilized flasks. Yeasts of the same type as Saazer yeast are not suitable for the estimation of total fermentable matter, but either high or low fermentation yeasts can be employed, even though a yeast of an opposite character is ordinarily employed in the actual fermentation of the wort.

Chem. Ztg., **17**, 1473. B. B. R.

385. Bau, A., March, 1893.—*The employment of yeast in the determination of fermentable substances* (*Ueber die Verwendung der Hefe zur quantitativen Bestimmung gährfähiger Substanzen*).

The liquid under examination should be thoroughly sterilized and the yeast should only be employed in a pure culture. The liquid must also be completely fermented. If, for example, a yeast is employed which only at first ferments dextrose and afterwards other hexoses, the results will be incorrect if fermentation is stopped prematurely. When fermentation is completed, the fermentation flask should not vary in weight during twenty-four hours, nor should fine, white foam form on the liquid, thus indicating the presence of CO_2.

Chem. Ztg., **17**, 392. B. B. R.

C.—Acids.

386. Prior, E.—*Separation and estimation of acids in beer, wort, malt, etc.* (*Trennung und Bestimmung der Säuren in Bier, Würzen, Malz u. dergl.*).

The author notes that he has already shown that a considerable part of the acidity of beer and worts is due to the presence of acid phosphates, and has given a process for the separation of these acid salts. The process now described provides for the estimation of fixed and volatile acids as well as the acidity due to the acid phosphates. To determine volatile acids 50 cc. of beer are distilled in a partial vacuum, water being admitted into the distillation flask at intervals, and the distillation being continued about three to three and one-half hours. The distillate is titrated with $\frac{N}{10}$ soda. To the distillation residue, which should not exceed 16 to 20 cc. in bulk, 75 cc. acid-free absolute alcohol are added, and then ether by degrees until the volume amounts to 500 cc., rotating the flask constantly. After standing about twenty-four hours it is filtered and the residue washed with a mixture consisting of 85 cc. ether and 15 cc. alcohol. After expulsion of ether and alcohol from the filtrate, water is added and the solution titrated. The acid phosphates on the filter are dissolved in water and also titrated with $\frac{N}{10}$ alkali. In a separate portion of the beer the total acidity is also determined. Phenolphthalein is employed as indicator, and for acid phosphates a special solution is prepared which contains a small amount of free alkali.

Bay. Brauer. Jour., 1892; Chem. Ztg. Rep., **16**, 296.

D.—Adulterants.

387. Elion, H.—*Detection and estimation of salicylic acid in beer, etc.*

Method depends upon agitation of acidified liquid with ether, the agitation of the latter with a little water alkaline with KOH, neutralization of this aqueous solution by HCl, and test of the residue by ferric chlorid, repeating the process of separation if necessary to secure a residue sufficiently free from other matters. To estimate it after securing it all in aqueous solution as above, the solution, evaporated nearly to dryness, is acidified slightly with H_2SO_4, bromin water added in excess, then KI and a little starch paste, and sodium sulphite until color is discharged; the tribromphenol is distilled over in a current of steam, separated by agitation to ether, this evaporated, and the residue dried over H_2SO_4 and weighed.

Trav. Chim. d. Pays-Bas., **7**, 211; Ztschr. anal. Chem., 1892, 96; Rev. internat. falsificat., **6**, 137. W. F.

388. GANTTER, F., May, 1892.—*The detection of saccharin in beer* (*Ueber die Brauchbarkeit der Fluoresceïnreaction zum Nachweis von Saccharin im Bier*).

The reaction for saccharin, recommended by Börnstein, and which consists in the formation of fluoresceïn by heating in the presence of concentrated sulphuric acid and resorcin, has, in a number of tests, indicated the presence of saccharin in beer when no saccharin was present, as shown by the absence of a sweet taste in the specially treated extract. Experiments showed that hop resin and even ordinary colophony gave the same reaction as saccharin with sulphuric acid and resorcin. The sweet taste is recommended as the surest test for saccharin, the liquid to be tested being evaporated to small bulk and, after addition of a drop of HCl, a large amount of alcohol poured in. The clear alcoholic solution is evaporated down and the residue agitated with ether; the ethereal solution is also evaporated and the residue treated with water, and this aqueous solution is concentrated to small bulk. In this residue, saccharin can be readily detected by its characteristic sweetish taste.

Ztschr. anal. Chem., **32**, 309; Jour. Chem. Soc., **64**, A, 504. B. B. R.

389. HAIRS, EUG.—*Determination of saccharin in the presence of salicylic acid* (*Recherche de la saccharine en présence de l'acide salicylique*).

Author notes similarity of behavior of these two substances toward ordinary solvents, and the inefficiency of ordinarily characteristic tests for each in the presence of the other. He proposes to treat purified mixture with bromin water, precipitating and removing the salicylic acid as bromosalicylic acid, which is scarcely at all soluble in water.

Jour. Pharm. d'Anvers; Rev. internat. falsificat., **7**, 24, 25. F.

DAIRY PRODUCTS.

MILK—GENERAL.

390. HITTCHER, DR.—*Methods for milk analysis and their practical application* (*Die verschiedenen Milchuntersuchungsmethoden und ihre Verwendung für die Praxis*).

Soxhlet's areometric method and the lactocrite are discussed at some length, and brief mention is made of Gerber's, Babcock's, Thörner's, and Lindström's methods.

The averages of fat tests made by the author from day to day agree fairly well with single tests made on composite samples preserved from four to six weeks with potassium bichromate, —0.1 gram for about 250 cc. of milk. The author, however, does not recommend composite sampling for the dairy.

Milch-Ztg., **22**, 320, 339. A. L. W.

391. RICHMOND, H. D., 1893.—*Accuracy in the analysis of dairy products.*—(*Part 1, milk analysis*).

Fat.—The author after trying several methods reached the following conclusions:

(1) That ether, petroleum ether, or chloroform, are equally effective solvents, if sufficient time be allowed for extraction. Ether is the most convenient as it extracts the fat most rapidly.

(2) That an accurate estimation can be obtained only if the milk solids are subdivided, either by the use of a paper coil free from matter soluble in the solvent, by evaporation of the milk or by some medium which can be very finely divided, or by extraction from a solution in which everything except the fat is dissolved (as in the Werner-Schmid process).

(3) That if ether is used as a solvent it must be pure and anhydrous.

(4) That the weight of fat obtained in the analysis may be taken to represent without appreciable error the amount originally existing in the milk.

(5) That in order to minimize the chance of error, a blank experiment should be carried out, following the conditions of the actual experiment as far as possible.

(6) That it is necessary to secure a true average sample of milk.

Total solids and water.—The Babcock method is adopted as the most accurate. It may possibly give too high results for total solids and too low results for water.

Adulteration of milk.—Genuine milks never furnish an ash of less than 0.7 per cent, while the majority of watered milks gave an ash below that figure. The nitrogen in genuine milks does not fall below 0.5 per cent. The percentages of nitrogen and insoluble ash are nearly the same in milk. For the detection of nitrates, the diphenylamin test is recommended. The author's method of procedure is as follows: Curdle the milk by heating nearly to 100° C.; add a small amount of acetic acid, and filter the serum. Place a little diphenylamin in a porcelain basin, and add 1 cc of pure sulphuric acid, and allow a few drops of the serum to trickle down the sides of the basin over the surface of the acid. If nitric acid be present a blue coloration is developed in ten minutes around the edges.

[From analyses of milk made in New Jersey we are led to take issue with the author's statements. We have found that genuine Holstein-Friesian milk contains considerably less than 0.7 per cent ash and 0.5 per centnitrogen. N. J. Exp. Sta. Bull., 77, p. 19.]

Milk sugar.—Vieth's modification of Wiley's method of polarization gives results which agree well with the gravimetric method of estimation by Fehling's solution.

Albuminoids.—The author regards the albuminoid precipitated by a saturated solution of magnesium sulphate as casein (including globulin), and the further precipitation by tannin or phosphotungstic acid in the filtrate from the casein as albumen. If he has reason to suspect that from some change in the milk albumoses are present he separates the albumen by heating after slight acidification and precipitates by tannin. He regards the precipitate as albumoses, but the determination is not very exact. The precipitate produced by adding copper sulphate to the neutral milk he regards as casein and albumen and designates this total proteids. He assumes that both casein and albumen contain 15.7 per cent nitrogen.

Jour. Am. Chem. Soc., **15**, 579–591; Chem. Centralbl., 1894, i, 233. J. P. S.

392. Sartori, A., 1893.—*The discovery of sulphur in cow's milk* (*Schwefel in Kuhmilch*).

Chem. Ztg., **17**, 1070; **17**, 1138; Exp. Sta. Record, **5**, 343. J. P. S.

393. Herz, F. J., 1893.—*Recent contributions on the chemistry of milk and its products* (*Ueber Neuerungen auf dem Gebiete der Chemie der Milch und der Erzeugnisse*).

Chem. Ztg., **17**, 1547–1551. J. P. S.

394. Meillère, 1894.—*The analysis of milk* (*Analyse du lait*).

Jour. pharm. chim., **29** (5), 153–155; Chem. Centralbl., 1894, **1**, 606, 607. J. P. S.

Milk.—Sampling, etc.

395. Siedel, Johs., and Teinann, H.—*Milk sampling; frequency requisite.*

Authors, as result of daily tests upon a herd of 20 cows, have determined upon weekly sampling as requisite.

Milch-Ztg., 1892, 34; Centralbl. Agrik. Chem., **22**, 209. W. F.

396. Alén, J. A.—*Preservation of composite milk samples.*

Author recommends a little potassium bichromate (0.5 gram) to be added to the sample jar, which is to be shaken daily, just after addition of the fresh daily subsample, carefully closed, and kept not higher than 15° C. If 20° C. is maintained, clumps of cream rise that are afterwards difficult of subdivision. Analyses show excellent preservation.

Kgl. Landtbruks-Akademiens Handlingar, 1892, 54-61; Centralbl. Agrik. Chem., **21**, 549, 550; Jour. Chem. Soc., **64**, ii, 308. W. F.

397. Patterson, H. J., December, 1892.—*The effect of the keeping of milk upon its fat contents.*

From a number of duplicate tests of milk and cream kept for various times and until they had attained various degrees of sourness, the author concludes that the results obtained by the Leffmann-Beam method would not differ materially, whether the milk or cream were sweet or sour, provided the sample was very carefully drawn.

Agric. Science, **6**, 553–556. W. F.

398. Davenport, B. F.—*Note on milk analysis.*

To restore to the emulsion any small quantity of butter which may have been churned out, addition of 1 per cent of ether together with thorough shaking is recommended.

Jour. anal. Chem., **6**, 480. A. L. W.

Milk.—Formulæ for Computation of Solids, etc.

399. Babcock, S. M.—*Babcock's formulæ for milk calculation.*

Gives tables showing percentage of solids not fat, corresponding to lactometer readings from 17° to 40° and 0 to 6 per cent of fat, and the following simplified formulæ:

$$\text{Total solids} = \frac{L + 0.7f}{3.8} + f,$$

and

$$\text{Solids not fat} = \frac{L + 0.7f}{3.8},$$

in which L equals reading of Quevenne lactometer at 60° F., and f equals per cent of fat.

The accuracy of the last formula is but slightly impaired by simplifying to

$$\text{Solids not fat} = \frac{L + f}{4}.$$

Results by these formulæ are within 0.1 per cent of those by more complicated formulæ. Rule given for making correction.

Wis. Sta. Rept. for 1891, 292–307; Exp. Sta. Record, **4**, 189. E. W. A.

400. Vieth, G.—*Fleischmann's and Hehner-Richmond formulæ for milk fat computation.*

Author finds that both give excellent results compared with Adams method determination, the difference being by the former formula, 0 — 0.4 per cent in whole milk and —0.1 to 0.2 in skim milk; by the latter formula, only —0.1 to +0.2.

Chem. Centralbl., 1892, i, 903; Centralbl. Agrik. Chem., **22**, 211. W. F.

401. Richmond, H. Droop.—*A rapid method of milk analysis.*

If the density of the milk be taken before and after filtration, the density of the milk after filtration less 1, divided by 0.004 very fairly represents the solids not fat, and the difference between the two divided by 0.0008 the fat. Results of 5 determinations given; greatest difference between the observed and calculated figures of solids not fat, 0.13 per cent, and for fat, 0.4 per cent.

Analyst, **17**, 50. F. W. W.

402. Richmond, H. Droop.—*The relation between specific gravity, fat, and solids not fat in milk.*

Comparison of 328 determinations of the fat content in milk, as found by direct analysis and calculated by the author's "milk scale;" the average difference within each group of 1 per cent varies from +0.021 with from 1 to 2 per cent to —0.046 with from 4 to 5 per cent of fat; total average difference —0.005 per cent. The following revised formula is given: $T - 0.254\ G = 1.164\ F_1$, and $F = F_1 - 0.2$ (solids not fat $- 8.87$).

Analyst, **17**, 169–171. F. W. W.

403. Nisius, J.—*Formula for calculating the specific gravity of the solids of milk from the per cent of fat in the solids (Ueber die durch die Giltigkeit der Fleischmann'schen Formel bedingte Beziehung zwischen dem spezifischen Gewicht und dem prozentischen Fettgehalt der Trockensubstanz der Milch).*

The author derives from Fleischmann's formulæ the following: $m = \frac{2665}{1665 + 12p}$ where m = sp. gr. of the milk solids and p = per cent of fat in the milk solids. Tables are given for obtaining p from m and m from p. These values are of use because they are not changed by the dilution or concentration of the milk.

Milk-Ztg. **22,** 272. A. L. W.

404. RICHMOND, H. DROOP.—*The relation between specific gravity, fat, and solids not fat in the milk of the gamoose.*

The formulæ T (total solids) $=27\frac{G}{D}+1.191\ F$, and protein $P=2.8\,\mathrm{T}+2.5\ A-3.33\ F-.7\frac{G}{D}$, were found to give satisfactory results.

Analyst, **17,** 5, 6. F. W. W.

405. HERZ, JOS.—*Determination of adulteration of milk by formulæ (Les falsifications du lait fixees par la comparaison avec l'épreuve de l'etable).*

Author defends the efficiency of the formulæ issued by the Memmingen milk station (Mitth. milchwirthschaftlichen Vereins in Algäu, 1892, Beilage zu Nr. 11) for the estimation of the extent of adulteration of milk by dilution with water and by stripping.

Rev. internat falsificat., **7,** 130-132. F.

MILK.—ESTIMATION OF SOLIDS.

406. RICHMOND, H. DROOP.—*The estimation of total solids in milk.*

Author uses the following method: About 3 grams of asbestos (of best quality) are placed in a platinum basin and ignited in a muffle; after weighing the basin 5 grams of milk are added and dried on a water bath for about two hours; it is then left in a water oven at about 98° for twelve hours or more (usually over night). The residue serves excellently for the purpose of ash determination.

Analyst, **17,** 225-227; Exp. Sta. Record, **4,** 584. F. W. W.

407. ELLIS, W. H., December, 1892.—*Official milk analysis in Canada.—Changes upon drying in air.*

In the course of milk control work, the writer studied the differences obtained in total solids, solids not fat, fat by differences and fat by Werner-Schmidt method, using a specially arranged drying oven, for description of which see original.

[The essential feature consists in placing the asbestos-filled drying tubes in copper tubes running horizontally through the oven, and closed at both ends, but connected with a central parallel tube, through which gas is passed, by vertical tubes at each end; this central tube is divided so that the gas current must pass through the drying tubes.]

It appears from these experiments that by long drying at the temperature of the water oven, the milk solids undergo changes by which the total solids increase in weight 1.8 per cent, the portion insoluble in gasoline increases 4.3 per cent, while the fat, determined by difference loses 4.5 per cent, a portion becoming insoluble in gasoline. MacFarlane's asbestos method, with drying in purified coal gas, is affirmed to be the simplest, most rapid, and one of the most accurate gravimetric methods for milk fat. It gave, on the average, about 0.2 per cent less than the Adams paper method.

Jour. Am. Chem. Soc., **14,** 363-368. W. F.

408. KINNEAR, J. B.—*Estimation of solids and fat in milk.*

About 1 gram of milk is poured onto a thin, flat, weighed glass plate about 2½ inches in diameter, and after weighing, is dried in a hot-air chamber, being loosened from the plate before it is quite dry. The dry solids are weighed and transferred to a half-ounce, weighed, stoppered bottle; 3 or 4 cc. of ether are added, the stopper tied down, and the bottle immersed in warm water for a few minutes, shaken for one minute, cooled and weighed. As much of the clear solution as possible is poured into a weighed watch glass, the stopper replaced, and the bottle again weighed, as well as the fat on the watch glass. From these data the total fat is calculated.

Chem. News, **68**, 1, 2; Jour. Chem. Soc., **64**, A, 559; Chem. Ztg., **17**, Rep., 198; Exp. Sta. Record, **5**, 104. J. W. F.

MILK.—ESTIMATION OF FAT.

A.—GRAVIMETRIC METHODS.

1. Paper process.

409. RICHMOND, H. DROOP.—*Fat extraction and fat calculation in milk analysis.*

A discussion of the differences in the paper and plaster methods of milk analysis.

Analyst, **17**, 48–50. F. W. W.

410. VIETH, P.—*The paper process of milk fat determination.*

Author finds that filter paper retains some of the fat as well as the protein of the milk filtered through it, and regards it as an indication favorable to the Adams process.

Chem. Centralbl., 1892, i, 903; Centralbl. Agrik. Chem. **22**, 211. W. F.

411. LIEBERMANN, LEO, and SZÉKELY, S., March, 1893.—*New method for determining milk fat (Neue Methode zur Bestimmung des Feltgehaltes der Milch).*

In a high glass cylinder add to 50 cc. of milk 5 cc. KOH solution (1.27 sp. gr.); agitate strongly after inserting stopper; add 50 cc. petroleum ether (sp. gr. 0.6631, b. p. 60° C.) [authors claim that ordinary ether dissolves more than fat], an emulsion being formed; then add 50 cc. of 95.8 to 96 per cent alcohol and again agitate. The petroleum ether separates. Repeat this three or four times, allowing time for the separation. Then determine the fat in an aliquot of the petrolic solution of fat by evaporation on a water bath and drying at 110° to 120° C.

Ztschr. anal. Chem., **32**, 168–173; Rev. internat. falsificat., **6**, 156, 157, **7**, 45, 46; Exp. Sta. Record, **4**, 778; Jour. Soc. Chem. Ind., **12**, 717; Ber. chem. Ges., **26**, R. 505; Milch-Ztg., **22**, 803, 804. W. F.

2. Weiss' method.

412. WEISS.—*Determination of fat content in milk (Bestimmung des Fettgehaltes der Milch).*

In a flask of 300 cc. capacity are brought 30 grams of milk. To this are added 3 grams soda solution (15 per cent) and, by degrees, 60 grams of petroleum benzine, shaking frequently until a homogeneous mixture is obtained. Next add 20 grams of alcohol, shake well, and set aside. After twenty-four hours three well-defined layers have formed, the uppermost of which contains the total fat in solution. An aliquot part of this liquid is taken, evaporated first at about 50°, and finally heated at 100° C. until a constant weight is obtained. From the weight thus found the total fat content is calculated.

Pharm. Ztg., **38**, 87; Chem. Ztg. Rep., **17**, 42; Chem. Centralbl., 1893, i, 589. B. B. R.

413. WEISS.—*Fat determination in milk (Zur Bestimmung des Fettgehaltes der Milch).*

The criticisms of Strassman on this process are referred to, and the following further conditions to be observed are given:

1. For pouring off the benzine fat solution one should wait until the settling is complete (about twelve hours).
2. Thorough shaking is necessary.
3. The heating of the fat is continued to a constant weight, since the last portion of benzine is expelled with great difficulty. The heating can be conducted gently for some time at 110°.
4. In measuring, care should be observed as to temperature, on account of the variability in volume of the benzine.

Pharm. Ztg. **38,** 258; Chem. Ztg. Rep., **17,** 134; Chem. Centralbl., 1893, ii, 159; Jour. Chem. Soc., **64,** A, 601. B. B. R.

414. LANG.—*Weiss' method for determination of fat in milk* (*Zur Bestimmung des Fettgehaltes in der Milch nach Weiss*).

The process of Weiss requires for its execution too great an amount of time, and preference is given to Liebermann and Szekely's method, which depends upon the same principle, though a different proportion of reagents is used. By proceeding as follows satisfactory results were secured: 50 cc. milk were mixed with 5 cc. potash solution (sp. gr. 1.27) and to this were added 50 cc. of petroleum ether of 0.663 sp. gr. After addition of 50 cc. alcohol (95.8 to 96 per cent), the separation of the ether-fat layer takes place readily. The mixture is next shaken two or three times, allowing the settling of the ether after each agitation. Finally, 20 cc. of the ether-fat layer is removed by a pipette, evaporated in a small flask on the water bath, and dried about an hour at 110° to 120° C.

Pharm. Ztg., **38,** 219; Chem. Centralbl., 1893, i, 960. B. B. R.

415. STRASSMAN, 1893.—*Fat determination in milk according to Weiss* (*Fettgehaltes-Bestimmungin der Milch nach Weiss*).

Reference is made to the method described by Weiss in Pharm. Ztg., **38,** 87, and the results of several practical tests of the method are reported. These results indicate that for accuracy this method does not compare with that of Soxhlet's, with Pfeiffer and Adams' modification. For practical purposes it is too complicated and tedious; for scientific work it is too uncertain. Results of determination by this method neither agree with check tests, nor with results obtained by the lactobutyrometer and by Soxhlet's process.

Chem. Ztg., **17,** 288. B. B. R.

416. RICHMOND, H. DROOP.—*The Werner-Schmid method of milk analysis.*

Author describes the modification used in the laboratory of the Aylesbury Dairy Company, London. Five cubic centimeters of milk are emptied into a stopped tube (Horsley tube) and diluted with 5 cc. of water. About 11 cc. of strong HCl are added, and the whole boiled over a naked flame till the fat forms a clear layer on the top. The tube is then cooled, 25 cc. of ether added, and the whole well shaken and allowed to settle, which happens practically instantaneously. As much as possible of the ether is pipetted off, and a further quantity added, and the treatment repeated three times. The ether is evaporated and the fat weighed. Results with this and Adams' method agree practically absolutely, according to the experience of the author.

Analyst, **18,** 53, 54. F. W. W.

417. PINETTE, J.—*Estimation of fat in milk by Schmid's method.*

The author heats 10 cc. of milk with 10 cc. of hydrochloric acid until the fat is liberated and the albumen nearly dissolved; then dissolves the fat in a mixture of ether and light petroleum, makes up to a definite volume, removes an aliquot, evaporates, dries, and weighs the fat.

Chem. Ztg., **15,** 1833; Jour. Chem. Soc., **62,** 1134. J. W. F.

B.—VOLUMETRIC METHODS

1. Babcock's method.

418. BABCOCK, S. M.—*Notes on the Babcock milk test.*

A reprinted description of the test, with some additional suggestions and reference to its application in testing cream, composite samples, etc.

Wis. Sta. Bul. No. 31; Expt. Sta. Record, **4,** 194. E. W. A.

419. SHUTT, F. T.—*On the Babcock method of milk analysis.*

Chem. News, **64,** 4; Analyst, **17,** 227–229. F. W. W.

420. Ahlborn, E., 1893.—*Ahlborn's machine for the Babcock milk test (Die Ahlborn'sche Ausführung des Babcock'schen Fettbestimmungsapparates).*

Milch-Ztg., **22**, 610–612. J. P. S.

421. Heinrich, R.—*Trials of the Babcock test (Vergleichender Prüfungen über die Zuverlässigkeit der Milchfett-Bestimmungen durch das Babcock'sche Verfahren).*

Reports comparisons of this test with the gravimetric test and the lactocrite. The result was very favorable to the Babcock test when the machine was run at a speed of 1,500 revolutions per minute. It was found to be cheaper, simpler, and more convenient than the lactocrite or Soxhlet's areometric method.

Molk. Ztg., 1893, 37, 38; Expt. Sta. Record, **4**, 277, 278. E. W. A.

422. Stokes, A. W.—*Estimation of fat in milk by the Babcock method.*

Author gives 12 comparative analyses of milk by Babcock's method and calculated or Schmid and Adams's method; the differences range from 0 to 0.5 per cent; average difference, 0.21 per cent. Results unsatisfactory on the whole. Author concludes that "though not a good laboratory process, the Babcock method is the best, cheapest, simplest, and most rapid dairy method of determining fat in milk."

[Note.—Too slow whirling and too strong H_2SO_4 most likely account for the poor results obtained; the bottles were whirled "at a rate of from 400 to 500 revolutions per minute; the fat in the tube is not the usual light-yellow butter fat, but a thick, brownish mass, often quite black."]

Analyst, **17**, 127–130. F. W. W.

423. Embrey, G.—*The Lister-Babcock milk tester, with some suggestions for extending its use.*

Author reports a number of comparative results with this and Adams's or calculated method; finds results with Babcock's method too low, and corrects by adding 4 per cent to compare it with "calculated fat," and 3 per cent to compare it with Adams's method. No fat globules found in the white sediment of selenite at the bottom of the test bottles. The low results claimed to be due to inaccuracy of graduating tubes.

[Note.—The calibration of tubes not shown to be faulty; in most tubes brought on the market in this country no errors of analysis like those found by the author could be accounted for by incorrect graduation. Results are most likely due to insufficient whirling, as the bottles were only whirled 600 revolutions per minute, instead of from 700 to 1,200, according to the diameter of the wheel which carries the bottles, as given in the directions. In small hand machines the error of too slow whirling is very common; every machine ought to be tested as to the proper speed of whirling before results are depended upon, especially where the diameter of the drum is rather small this is very important; if tests of the same milks whirled at a higher speed than that ordinarily used give the same results as before, the test is correct; when results differ, those obtained by the more rapid whirling (the highest ones) are taken.]

Analyst, **18**, 118–123. F. W. W.

2. Leffmann and Beam method.

424. Leffmann, H., and Beam, W.—*A rapid and accurate method of determining fat in milk.*

Fifteen cubic centimeters of milk are mixed in a test bottle with 3 cc. of a mixture of equal parts of fusel oil and strong HCl; the bottle is then filled up to the neck with strong H_2SO_4, whirled in a centrifugal machine from one to two minutes, and the length of the column of fat in the graduated portion of the bottle ascertained. Comparative analyses by this and the Adams method given.

[Note.—This is the same method as was published by Vt. Exp. Station in Bull. 21 about three years ago, and known under the name of the Beimling test.]

Analyst, **17**, 83, 84. F. W. W.

425. Hehner, O.—*Note on the Leffmann-Beam method of determining fat in milk.*

Seven analyses of milk by this and by Adams's method given; greatest difference, 0.09 per cent (higher by Adams's method).

Analyst, **17,** 102, 103. F. W. W.

426. Leffmann, Henry, and Beam, William.—*Authorship of the Leffmann-Beam or Beimling milk fat method.*

Authors claim that the chemical part of the method was devised by them, so that it should be known as the Leffmann-Beam method.

Agric. Science, **6,** 464. W. F.

427. Richmond, H. Droop.—*Leffmann-Beam method of fat estimation in milk (Part I).*

A critical investigation of the method; the author gives a large number of comparative analyses by this and standard methods, which usually agree very well; the sources of errors to which the method is open are enumerated and discussed; the total error due to the causes will not, according to the author, exceed 0.1 per cent with ordinary care. The method is recommended for commercial and dairy control purposes, but is not considered a proper laboratory method.

Analyst, **17,** 144–152. F. W. W.

428. Richmond, H. Droop.—*The Leffmann-Beam method for the estimation of fat in milk (Part II).*

A continued investigation of the method (see above, also Analyst, **17,** 144–152). Author finds it necessary to multiply readings by 1.08 in order to obtain the actual per cent of fat in the milk; he concludes that amyl hydrogen sulphate is not dissolved in the fat. The actual factor connecting readings and percentages of fat must be worked out for each bottle, as it will not do to trust the graduations, even if the absolute volume is checked. When bottles are once graduated, author finds the method very accurate and reliable. Creams may be roughly analyzed by its means, but do not give as satisfactory results as milk.

Analyst, **18,** 130–134. F. W. W.

429. Richmond, H. Droop, and Boseley, L. K.—*Leffmann-Beam method for determining fat in milk.*

Analyst, **19,** 72; Chem. Centralbl., 1894, i, 843, 844.

3. *Patterson's method.*

430. Patterson, L. G.—*Patterson's method of testing milk.*

Modified Beimling method, using amyl alcohol instead of hydrochloric acid and amyl alcohol.

Miss. Sta. Bul. No. 21; Exp. Sta. Record, **4,** 267; Jour. Chem. Soc., **64,** ii, 252.

E. W. A.

4. *Lactocrite.*

431. Neumann, J.—*A new acid mixture for the lactocrite (Ueber die Bestimmung des Fettgehaltes der Milch vermittels der neuen Laktokritsäure.)*

The mixture consists of 100 volumes of lactic acid and 5 to 8 volumes of hydrochloric acid. The manipulation differs from that where the usual acetic acid mixture is used in two particulars: (1) The acid mixture is first measured out and heated on a boiling water bath before adding the milk, and (2) the whirling is carried on at 60° C. instead of 40° C. Results accurate.

Milch-Ztg., **21,** 625; Ztschr. angew. Chem., 1892, 733; Centralbl. Agrik. Chem., **21,** 840; Chem. Centralbl., 1892, ii, 760. A. L. W.

432. Hittcher.—*Fat determination by use of the lactocrite.*

Author states that in using the new acid reagent suggested by de Laval (hydrochloric and lactic instead of sulphuric and acetic acids), although he heated the mixture of milk and the reagent for fifteen minutes and used high speed, an opaque section of solidified fat was formed in the tube, necessitating correction. W. F.

Deutsch. Molkerei Ztg, 1892, 10; Centralbl. Agrik. Chem., **21**, 840.

433. ALÉN, J. C.—*Coloring of the fat column in fat determination by the lactocrite.*

Author proposes addition of alkanet to the acid mixture (sulphuric and acetic acid), and says that the fat will take up the color and give a sharp reading in consequence.

Tidskrift f. Landtmän, **13** (1892), 51; Centralbl. Agrik. Chem., **21**, 856.

W. F.

5. Lindström butyrometer.

434. *Lindström butyrometer—(Lindström-Butyrometer zur Bestimmung des Fetts in der Milch).*

A milk test similar to the lactocrite. The milk is first boiled in the test bottle with lactic acid, to which some sulphuric acid has been added. The fat is separated by whirling in a centrifugal machine, brought into the graduated neck of the bottle by adding water through an opening in the side of the test bottle, cooled until it becomes solid, and measured. The apparatus is figured.

Milch-Ztg., **21**, 496. A. L. W.

435. BACKHAUS and NEUMANN.—*Lindström butyrometer.*

The process is described and discussed. Results are given of numerous tests made with Lindström's apparatus, using the acid sent out by the makers and various acid mixtures. In the following table are given the widest and the average differences of these results from those obtained by chemical analysis:

	Widest+ difference.	Widest− difference.	Average difference.
Butyrometer acid	0.15	0.12	+0.06
C. P. lactic acid containing 8 per cent sulphuric acid	.07	.15	—.04
Acetic acid containing 5 per cent sulphuric acid	....	.30	—.14
Mixture 100 volumes lactic acid, 100 volumes acetic acid, and 15 volumes sulphuric acid	.03	.07	—.01
Mixture 200 volumes lactic acid, 100 volumes acetic acid, and 25 volumes sulphuric acid	.06	.04	+.006

Milch-Ztg., **22**, 85. A. L. W.

6. Demichel's lactobutyrometer.

436. GRAFFENBERGER, L.—*Milk fat determination with Demichel's lactobutyrometer.*

As a result of tests, author recommends the addition of two drops of caustic potash, the use of 91 or 92 per cent alcohol, the separation of the fat by standing ten minutes in water at 40° C., and reading off after standing for half an hour at 20° C. Individual determinations differed by 0.3 per cent, while with the Soxhlet areometric apparatus the difference was 0.20.

Landw. Vers.-Stat., **41**, 43–54; Centralbl. Agrik. Chem., **21**, 839, 840; Chem. Centralbl., 1892, ii, 760; Milch. Ztg., **21**, 809. W. F.

7. Thörner's method.

437. THÖRNER, W., 1892.—*Estimation of fat in milk.*

Ten cubic centimeters of milk are placed in a tube of special shape, and saponified by adding 1.5 cc. of alcoholic potash (16 grams per liter) and placing the tube in the water bath for two minutes. One cubic centimeter of strong acetic, sulphuric, or other acid is added to separate the fatty acids, the tube is again heated, then centrifugalized one to four minutes at 2,000 revolutions per minute, heated to 100° and the volume of the fatty acids read off.

[Essentially Short's process without the centrifugal.—Ed.]

Molk. Ztg., **2,** 1892, No. 1; Chem. Centralbl., 1892, ii, 429; Jour. Chem. Soc., **64,** ii, 101; Centralbl. Agrik. Chem., **22,** 416, 417; Milch-Ztg., **21,** 632; Chem. Ztg., **16,** 1101. J. W. F.

438. Lange, Gerhard.—*Thörner's milk test (Zur Anwendung der Centrifuge im Laboratorium).*

Thörner's test is described as a modification of Short's method.

Ztschr. angew. Chem., 1892, 488. A. L. W.

439. Krüger and Schwarz.—*Thörner's milk-fat method.*

Authors find that the average result by this method, using the centrifugal apparatus, is 0.021 higher than that by Soxhlet's process.

Molk. Ztg., 1892, 13; Centralbl. Agrik. Chem., **22,** 417. W. F.

440. Greff, H., 1893.—*Some new apparatus for use in the Thörner method of determining fat (Einige neue Hilfsapparate für die Fettbestimmungen nach der Thörner'schen Methode).*

Milch-Ztg., **22,** 608–610; Chem. Centralbl., 1894, i, 117, 118. J. P. S.

8. *Modified Schmid method.*

441. Leze and Allard.—*Determination of fat in milk products.*

The method is founded on the same principle as that of Schmied. The milk is heated with strong HCl for several hours at 25–30° C., afterwards neutralized with ammonia, warm water is added and the volume of fat read off in the graduated neck of the bottle in which the reaction is conducted.

Comptes-Rendus, **113,** 654; Chem. Ztg.; Analyst, **17,** 55–57. F. W. W.

9. *Acidobutyrometric method.*

442. Gerber, N., December, 1892.—*The acid-butyrometric method for fat estimation (Die Acid-Butyrometrie als Universal-Fettbestimmungs Methode).*

The apparatus described and illustrated provides for the estimation of the fat by the centrifugal process, the amount of fat being read off from the graduations on the tube employed in the determination.

The milk is first mixed with a small proportion of amyl alcohol, and then about an equal volume of acid, shaking the mixture thoroughly. The tube is then placed in the centrifugal machine, and after a rotation for two to two and one-half minutes, the separation of fat is complete. By a slight modification of the process, the fat in cream, butter, etc., can be determined.

[Essentially the Leffmann-Beam method.]

Chem. Ztg., **16,** 1839; Ind. Lait., 1892, 397, 413; 1893, **1,** 43; Analyst, 137, 138.

B. B. R.

C.—AREOMETRIC METHOD (SOXHLET'S).

443. Fischer, Sartori, Runschke, and Butler.—*The examination of milk (Mittheilungen aus dem chemischen Untersuchungsamte der Stadt Breslau). [Soxhlet process.]*

The authors have abandoned the areometric method of Soxhlet for the estimation of fat, and have returned to the use of Marchand's process. The chief objections to Soxhlet's method are that the ether fat layer in Soxhlet's apparatus does not separate completely, or only with great difficulty, without the aid of a centrifugal; and that the scale of the spindle does not permit of the estimation of fat in very rich milks (above 5.12 per cent fat).

Jahresber. chem. Untersuch. Breslau, 1891–'92; Chem. Ztg. Rep, **16,** 349.

B. B. R.

444. Janke, L. 1894.—*Some new methods for the determination of milk fat, especially some comparative results by the Soxhlet areometric and the Gerber butyrometric methods (Ueber einige neue Milchfettbestimmungsmethoden, insbesondere über vergleichende Untersuchungen mittels des Soxhlet'schen aräometrischen und des N. Gerber'schen butyrometrischen Verfahrens).*

Fifty-six determinations gave an average difference between the two methods of less than 0.1 per cent; in all cases but nine the Gerber method gave the higher results. Gerber's method was found to be very satisfactory for chemical practice, and possesses the merits of accuracy and rapidity. The author obtained better results at a temperature of 60° in this method than at 70°, as previously recommended.

Forsch. ber. über Lebensmittel., **1,** 79–82; Chem. Centralbl., 1894, i, 526, 527.

J. P. S.

445. Albert, 1893.—*An apparatus for measuring out the ether and potassium hydrate used in the Soxhlet areometric method (Ein Hilfsapparat zum Abmessen von wasserhaltigem Aether und Kalilauge für den Soxhlet'schen aräometrischen Apparat zur Fettbestimmung in der Milch).*

Milch-Ztg., **22,** 461, 462. J. P. S.

446. Timpe, H., 1894.—*The Soxhlet areometric method for the estimation of fat (Ueber die Soxhlet'sche aräometrische Fettbestimmungsmethode).*

Chem. Ztg., **18,** 392–394; Chem. Centralbl., 1894, i, 844, 845; Ber. chem. Ges., **27,** Ref. 343. J. P. S.

447. Gräffenberger.—*Comparative determinations of fat in milk by the methods of Soxhlet, Schmidt, etc.*

Landw. Vers.-Stat., **43,** 247; Chem. Ztg., **18,** Rep., 16; Chem. Centralbl., 1894, i, 442.

Milk Sugar.

448. Gill, A. H.—*Note upon the estimation of milk sugar in milk.*

Author recommends the following method: 25 cc. of milk are mixed with 15 cc. of milk of alumina, and 0.5 cc. of 25 per cent acetic acid added, the mixture stirred, heated on water bath for five to seven minutes at 85° C.; 100 cc. of water are then added, and the mixture heated in boiling water for ten minutes, cooled, allowed to settle, decanted through a plaited filter (S. & S. 597) into a 500 cc. flask, being careful to bring as little precipitate as possible on the filter; the boiling and filtering are repeated three times, and the filtrate made up to 500 cc. and finally titrated by Fehling's solution, as usual.

The milk of alumina is prepared by precipitating 125 grams of ammonia alum with ammonium hydrate at boiling temperature, washing the precipitate by decantation, and making up to 1 liter.

Analyst, **17,** 36. F. W. W.

449. Bornträger, A.—*Influence of basic acetate of lead in the volumetric determination of milk sugar by the Fehling-Soxhlet method (Ueber den Einfluss welchen die Gegenwart von Bleiessig auf das Ergebniss der Titrirung des Milchzuckers nach Fehling-Soxhlet ausübt).*

When basic acetate is present too much milk sugar is required for complete reduction. The lead should be removed before titration by precipitation with sodium sulphate.

Ztschr. angew. Chem., 1892, 293; Ber. chem. Ges., **25,** Ref. 598; Chem. Centralbl., 1892, ii, 135. A. L. W.

450. Richmond, H. D.—*Effect of heating milk on specific rotation of milk sugar.*

Analyst, 1893, 141, 142; Exp. Sta. Record, **4,** 11.

451. Denigés, G.—*Recognition and determination of lactose in milk.*

Jour. pharm. chim., **27** [5], 413; Chem. Centralbl., 1893, i, 1092.

452. Denigés, G.—*Determination of lactose in milk* (*Dosage de la lactose dans les laits*).

Objection is made to clarification of milk, for polarimetric reading, by lead subacetate on the ground that it does not completely remove albuminoids, so that the readings are often 2° (saccharimetric) too high; and to that by mercuric acetate, in that the mercury attacks the brass mountings of the tubes, and also interferes with subsequent use of the solution with Fehling's reagent. He prefers the use of metaphosphoric acid, or, better, freshly prepared sodium metaphosphate. The latter is prepared by carefully heating ammonio-sodium phosphate in a platinum dish until it is completely fused and no longer evolves gas. Pour on a cool plate, break up, and keep in stopped bottle. Prepare a 5 per cent aqueous solution, by adding 5.7 grams of the finely divided salt to 50 cc. of boiling water; boil five minutes, when solution should be complete. Twelve per cent (0.7 gram) of the salt will have been converted to orthophosphate, leaving 5 grams unchanged. Immediately add 5 cc. of cold water, cool under jet of water, make up to 100 cc.

To analyze the milk add to 10 cc. of the milk 25 cc. of this solution, 60 to 70 cc. of water, shake, add 0.3 cc. of acetic acid (in woman's milk 0.5 cc. of HCl should be used), make up to 100, filter, reject the first few drops, and gather the clear fluid, which gives no albuminoid reaction with the tests of Millon, Tanret, and Eschbach, and which is clearly read. Even woman's milk, by repeated filtrations, gives a clear solution. HCl is to be preferred to acetic acid, except where adulteration by cane sugar is suspected. Use a 50 cm. tube in preference to a 20 cm.

Bull. Trav. Soc. Pharm. Bordeaux; Bull. Soc. Chim., **7** [3], 493–499; Rev. internat. falsificat., **6**, 190,191, 212, 213; Jour. Pharm., **27** [5], 413, 417; Jour. Chem. Soc., **64**, A, 248, 502. F.

Lactic Acid.

453. Thörner, W.—*Estimation of lactic acid in milk.*

Ten cubic centimeters of the sample are diluted to 30 cc. and mixed with 5 drops of a 5 per cent alcoholic solution of phenolphthalein. Decinormal soda is added until the liquid is pink. Should a milk take 1 cc. of the alkali, the author calls this 10° of acidity.

Chem. Ztg., **16**, 1469–1470, 1519–1520; Jour. Chem. Soc., **64**, ii, 250. J. W. F.

454. Trillich, H., October, 1892.—*Lactic acid estimation* (*Zur Milchsäure-Bestimmung*).

Issue is taken with Thörner in his statement (Chem. Ztg., **16**, 1469) to the effect that in lactic acid determinations the results would be scarcely affected appreciably by the employment of well water, previously boiled, in the dilution of the milk samples, previous to titration. This assertion of Thörner holds good only when the water contains no magnesium bicarbonate or only small quantities of it. With waters containing along with calcium carbonate a considerable amount of magnesium bicarbonate a strongly alkaline reaction is exhibited after boiling. Such waters can not be employed without making corrections for each acid determination in milk.

Chem. Ztg., **16**, 1565. B. B. R.

Proteids.

455. Carcano, L.—*Determination of nitrogen and proteids in milk and dairy products.*

Staz. Sper. Agr. Ital., **22**, 261; Chem. Centralbl., 1892, ii, 56.

Miscellaneous.

456. Thörner, W.—*Relation of cream content to butter content of milk.*

Chem. Ztg., 1892, 757; Chem. Centralbl., 1892, ii, 187; Exp. Sta. Record, **4**, 213.

457. THÖRNER, W.—*Method of examining milk for tuberculosis bacilli*

Chem. Ztg., 1892, 791, 792; Exp. Sta. Record, **4,** 214.

458. RICHMOND, H. DROOP.—*Action of some enzymes on milk sugar.*

Analyst, 1892, 222; Exp. Sta. Record, **4,** 584.

459. OKULITSCH, J., 1894.—*The question of the determination of specific gravity in sour milk* (*Zur Frage über die spez. Gewichtsbestimmung geronnener Milch*).

A further study of the method suggested by M. Weibull (Chem. Ztg., **17,** 1670, which see).

Milch-Ztg., **23,** 84. J. P. S.

460. WEIBULL, M., 1894.—*The estimation of fat in curdled milk* (*Ueber die Bestimmung des Fettes in geronnener Milch*).

The author arrives at the following conclusions from his results:

1. That by souring, milk loses a small amount of fat, which, however, during the first week, if kept in a closed flask and at a temperature of about 20 degrees, does not amount to more than 0.05 per cent. The loss found in skim milk is not greater, comparatively.

2. The curdled milk is best prepared for the estimation of the fat in the same way as for the estimation of the specific gravity. If a known amount of ammonia is added to the curdled milk and shaken, the amount of milk can be easily calculated from the total volume. From the amount of fat in the ammoniacal mixture the amount of fat in the milk can be easily obtained. The simplest way is to add exactly one-tenth ammonia, then by adding one-tenth to the fat found the real amount can be determined.

3. For exact analyses it is necessary to take into account the specific gravity and the adhesion of the milk. If the milk is not weighed, but measured, a special pipette must be used, or the proper correction made.

4. With the lactocrite, butyrometer, and other methods in which the milk is measured by a pipette of fixed volume, an almost constant error must be recognized. By a deduction of 0.05 per cent (in normal milk of about 3 per cent fat mixed with ammonia, as previously described) this error can be corrected.

Chem. Ztg., **18,** 926–928; Milch-Ztg., **23,** 413, 414. J. P.S.

461. WEIBULL, M., 1893.—*Can the specific gravity of curdled milk be accurately determined* (*Kann man das spez. Gewicht einer Milch die geronnen ist, genau bestimmen*)?

The author answers the question affirmatively, and suggests the following method: A known amount of ammonia (about one-tenth the volume of the milk) is added to the curdled milk and shaken until all is in solution. The ammoniacal solution is then accurately measured into a cylinder, the original volume of milk determined (the total fluid less the added ammonia), and the sp. gr. and temperature of the liquid determined in the usual way. The data are sufficient for the desired end. After several experiments it has been found that the contraction of the fluid from the addition of the ammonia is so slight as not to appreciably affect results. The following equation expresses the operation:

$$\text{V am} \times \text{Sp am} + \text{V m} \times \text{Sp m} = \text{V fl} \times \text{Sp fl},$$

where V am, V m, and V fl signify the volume of the ammonia, milk, and ammoniacal fluid, respectively; and Sp am, Sp m, and Sp fl the corresponding sp. gr. The specific gravity of the milk is here the undetermined quantity, and can be easily obtained by a simple calculation.

Chem. Ztg., **17,** 1670; Chem. Centralbl., 1894, i, 116; Exp. Sta. Record, **5,** 644, 645. J. P. S.

462. WEIBULL, M., 1894.—*Can the specific gravity of curdled milk be determined accurately* (*Kann man das spezifische Gewicht einer Milch, die geronnen ist, genau bestimmen*)?

After an extended use of the method suggested by the author (Chem. Ztg., **17,** 1670), he arrives at the following conclusions:

1. The specific gravity of normal milk, which is curdled, and has stood for one or two weeks, can be accurately determined by the method suggested.
2. With careful work the addition of about one-tenth of ordinary ammonia to thet milk causes an error of not more than 0.0005. The reason for this lies in the fact tha the very small contraction of the fluid compensates for the decreased content of dry matter caused by the curdling of the milk.
3. This contraction at all events predominates so much that the results are rather a little too high than too low.
4. For accurate results the ammonia should not be added in too great an excess.

Milch-Ztg., **23,** 247, 248. J. P. S.

463. SCHAFFER.—*The detection of goat's milk in cow's milk* (*Nachweis der Ziegenmilch in der Kuhmilch*).

The detection of the presence of goat's milk in cow's milk is proposed by obtaining an ethereal solution of the fat, having previously added an alkali to the milk. Goat's milk fat gives a clear, colorless solution in ether, while the fat of cow's milk, specially in summer, gives a deep yellow solution. The author claims that an addition of 20 per cent goat's milk can in this manner be detected. B. B. R.

Schw. Woch. Chem. Pharm., **31,** 58; Chem. Ztg. Rep., **17,** 67.

464. BABCOCK, S. M.—*Detection of adulterations in milk.*

Remarks on milk standards, use of lactometer, and milk calculation.

Wis. Sta. Bull. No. 31; Exp. Sta. Record, **4,** 193. E. W. A.

BUTTER.

A.—GENERAL.

465. JEAN, F.—*Observations on butter analyses.*

Rev. internat. falsificat., 1891, 65; Analyst, **17,** 117. F. W. W.

466. SCHRODT, M., and HENZOLD.—*Interpretation of results of butter analysis.*

Landw. Vers.-Stat., **40,** 299–309; Exp. Sta. Record, **4,** 92.

467. RICHMOND, H. DROOP.—*On gamoose butter.*

The results of the examination of winter and summer butter for saponification equivalent, insoluble fatty acids, iodin number, soluble acids, and Reichert-Wollny method, are given in the paper.

Analyst, **17,** 46.

468. SPAETH, EDWARD.—*Butter analysis* (*Ueber Butteruntersuchung*).

The article describes a form of apparatus for the estimation of water and fat in butter. The apparatus consists of a glass-stoppered weighing glass, with perforated top and bottom, and a glass boat of such size that it can be placed in the weighing glass. A film of asbestos is placed over the bottom of the weighing glass, and the boat filled to one-third its capacity with pumice stone. They are then dried and weighed together. Eight to ten grams of butter are then introduced into the boat and dried, first on the water bath for one-half hour, and then in the air bath at a temperature a little over 100° for two to two and one-half hours. The boat and contents are placed in the weighing tube and the whole weighed. The loss is water. The weighing glass, tube, and contents are then placed in a Soxhlet's extraction apparatus and extracted with ether four to six hours. The fat is determined directly after evaporating the ether, and the result may be controlled by drying and weighing the weighing glass and boat.

Ztschr. angew. Chem., 1893, 513; Vjschr. Chem. Nahr., 1893, **8,** 223; Jour. Chem. Soc., 1894, **66,** ii, 124. W. D. B.

469. LAYES, E.—*Analysis of butter.*

The author finds that the method of König and Hart is more accurate than other methods, and suggests a few modifications.

Arch. Pharm., **231**, 356–366; Jour. Chem. Soc., **64**, A, 602. J. W. F.

B.—OPTICAL EXAMINATION.

470. JEAN, F.—*Notes on the optical and chemical analysis of butters.*

The author replies to a number of objections raised against his method of applying Amagat and Jean's "oleorefractometer" to the detection of adulteration in butter. To the chief objection made, viz, that perfectly pure butters show deviations by this method much below the normal (— 30°), the answer is made that butters of this character have been found to be derived invariably from the milk of cows fed on oil cake, and the abnormal behavior of the butter is ascribed to the presence of unaltered oil in the milk. In such cases the oleorefractometer can not be relied on exclusively, but by the employment also of chemical tests along with it, it is of utility in examining doubtful samples. In impure butters the volatile acids are below the mean value, while butters containing oil derived from oil cake as feed are not perceptibly affected in this particular. A large proportion of butters coming under examination, however, can be readily classified by means of the oleorefractometer.

With a deviation of from 32° to 36°, the purity of the butters is doubtful; palm or cocoanut oil may be present; 31° to 29°, pure butters; 29° to 25°, purity doubtful, and volatile acids must be determined.

Monit. Scient., **6**, 91; Jour. Soc. Chem. Ind., **11**, 945; Rev. internat. falsificat., **8**, 133; Milch-Ztg., **21**, 677. B. B. R.

471. PIZZI, H.—*On the (so-called) Pennetier's method for butter analysis.*

(Microscopic examination by polarized light.)

Staz. sper. agr. Ital., **23**, 38; Analyst, **18**, 91, 92. F. W. W.

472. RICHMOND, H. DROOP.—*The microscopic examination of butter by polarized light.*

An abstract of papers published on this subject; author states that most English analysts have discarded the method.

Analyst, **17**, 193, 194. F. W. W.

473. RICHMOND, H. DROOP.—*Amagat and Jean's oleorefractometer.*

The instrument is recommended for preliminary examination of butters. Pure butter will have a refraction of 28 to 34. When results come over 34 or under 28, Reichert's method should be used; if refraction is 20 or under, the article must be adulterated at least 50 per cent.

Analyst, **17**, 221–222. F. W. W.

474. ZUNE, A. J.—*The use of the oleorefractometer in butter analyses (Sur l'oléoréfractomètre).*

The author discusses the usefulness of the Amagat-Jean oleorefractometer for the detection of butter adulteration and denies its delicacy and efficiency.

Rev. internat. falsificat., **6**, 12, 13. W. F.

C.—MELTING POINT.

475. MAYER, A., 1893.—*Concerning the method of determining the melting point of butter.*

Narrow tubes give lower melting and higher congealing points than wide tubes. Lower melting points are always obtained when the fat is warmed slowly than when it is warmed rapidly. By a slow warming is meant a rise of one degree in five minutes. To obtain good melting points one must be careful that the previous congelation was complete, for which in butter-like substances a cooling to about 10° below the actual congealing point is absolutely necessary.

Jour. Am. Chem. Soc., **15**, 661–664; Chem. Centralbl., 1894, i, 234. J. P. S.

476. **Reich, E.**, 1893.—*The melting test of butter* (*Die Butter-Schmelzprobe*).
Milch Ztg., **22**, 787–789; Exp. Sta. Record **5**, 724. J. P. S.

477. **Viollette, C.**—*Analysis of commercial butters* (*Analyse des beurres du commerce*).

The author finds that the specific gravity of butter $\left(\frac{100° \text{C.}}{4° \text{C.}}\right)$ is 0.86320–0.86425, that of margarin 0.85766–0.85865, and that in a mixture of butter and margarin the proportion of each may be estimated by the specific gravity of the mixture. A densimeter has been constructed, by the aid of which a butter may be examined in a moment after it is heated to 100°.

Compt. rend., 1893 **117**, 856–858; Jour. Chem. Soc., 1894, **66**, ii, 124. W. D. B.

D.—Water estimation.

478. **Delaye, Louis.**—*Notes on butter analysis.—Moisture determination and determination of volatile acids.*

Writer objects to use of 20 grams of butter for moisture determination because of danger of loss by spitting. He says 10 grams should be the maximum quantity used, and suggests the absorption of the butter upon a tared spiral of perfectly dry filter paper, which will lead to a large surface exposure and remove all possibility of spitting. He claims that with 90° alcohol for saponification the danger of formation of esters of the volatile acids is greatly increased, and recommends 70°, which will secure complete saponification unless paraffin adulterants be present. The saponification flask should be of a volume greater than 200 cc.

Bull. Soc. Roy. Pharm. Brux.; Rev. internat. falsificat, **6**, 63–65.

W. F.

479. **Allen, A. H.**—*Proportion of water in butter.*

Analyst, 1892, 107–109; Exp. Sta. Record, **4**, 95.

480. **Hofmeister, W.**, 1894.—*The water content of butter* (*Untersuchungen betreffs des Wassergehalts der Butter.*)

Milch-Ztg., **23**, 51. J. P. S.

E.—Fat estimation.

1. *General.*

481. **Holter, George L.**, July, 1892.—*A volumetric method for the determination of fat in butter.*

The method is a modification of the Babcock volumetric milk test. The bottle consists of two parts; the bottle proper, of 20 cc. capacity, with a narrow neck, like the Babcock bottle, but only 12 mm. long; the second part is a bulb-tube, ground to fit the neck of the bottle proper, to which it is attached by a rubber tube, and graduated above and below the bulb in such manner as to include the volume of the bulb in the graduation. Melted thoroughly mixed butter, about 9 or 10 grams, is transferred to the tared test-bottle, cooled under a bell-jar, and weighed; it is then remelted, 5 cc. of hot water added, and the bottle shaken thoroughly. Concentrated sulphuric acid is then slowly added from a burette, with constant agitation, until a dark pink color is assumed, 2 to 5 cc. of acid being requisite; whirl rapidly for one or two minutes in a Babcock centrifugal, to separate the fat. Connect the graduated tube, place the bottle in a beaker of hot water, 250 mm. deep, add hot water enough to transfer the fat to the neck of the test-bottle proper; again whirl in the centrifugal for one minute, transfer to the hot water beaker, add enough hot water to transfer the fat to the graduated tube, but not enough to place the lower meniscus in the bulb; then whirl slowly in the centrifugal for a short time, transfer the bottle to water at 60° C., and read. The volume of fat multiplied by 9, and divided by the weight of butter taken, will give the percentage in the butter.

Agric. Science, **6**, 312–316; Milch-Ztg., **22**, 408. W. F.

482. SWEETSER, W. S., September, 1892.—*A simple method for the determination of fat in butter.*

Ten grams of well-mixed butter are melted and washed into a 200 cc. flask with 76° benzine. After shaking well and cooling, the flask is filled to the mark with benzine. The contents are again well mixed, allowed to subside, and 40 cc. of the clear solution drawn off by a pipette into a weighed platinum dish containing a few grams of asbestos. The solvent is driven off at gentle heat, the residue dried for one to two hours at 100° C., and weighed. The curd and ash may be filtered off from the solution in the flask, thoroughly washed with benzine by decantation and in a Gooch crucible, and then weighed, ignited and weighed, as usual. The method gave slightly lower results, and somewhat more concordant than the "official" method.

Agric. Science, **6,** 406–408. W. F.

483. FAHRION, W., May, 1893.—*A new method for fat examination* (*Ueber eine neue Methode der Fettuntersuchung*).

Reference is made to the method proposed by Hazura for the examination of fatty acids, in which process the fat is treated with permanganate in an alkaline solution. Since in this process the lead salts of the unsaturated fat acids are first separated from the saturated by extraction with ether, this mode of procedure is very time consuming.

The author proposed the direct employment of potassium permanganate solution (5 per cent), this being added to the hot alkaline liquid after complete saponification has been effected. After filtration and acidulation of the filtrate, the precipitate thrown down is treated upon a linen filter with petroleum ether, by means of which the unchanged and unoxidized part of the liquid fat acids go into solution, while the oxidized products remain behind undissolved.

Chem. Ztg., **17,** 610. B. B. R.

2. Unsaponifiable matter.

484. MAUSBRIDGE, W.—*New method for the estimation of unsaponifiable matter in fats.*

After the mass has been decomposed with acid, subsequent to saponification, a convenient quantity is treated with 50 cc. methyl alcohol, saturated with light petroleum boiling at 43° C, and 50 cc. of the light petroleum is then added. The whole is heated to boiling in a reflux apparatus, and, after cooling slightly, neutralized with a measured quantity of $\frac{N}{2}$ potassium hydroxid and placed in a separating funnel. If the separation is imperfect a few cubic centimeters of boiling alcohol are added. The petroleum layer is separated, and washed by agitating with 50 cc. of water at 38° or with 50 per cent alcohol, and then with 40 cc. of 70 per cent alcohol. Distil and dry, and a second extraction with light petroleum removes the unsaponifiable matter.

Chem. News, **65,** 253–255; Jour. Chem. Soc., **62,** 1533. J. W. F.

485. HONIG and SPITZ.—*Examination of mixture of unsaponifiable and saponifiable fats* (*Examen de mélanges de graisses saponifiables et de graisses nonsaponifiables*).

More exact results are claimed in case of such mixtures if, after saponification, the unsaponifiable matters are removed by washing with petroleum ether. The details are so simple as to need no description, being such as are common in the separation of immiscible liquids. It is claimed that for approximate estimation of the unsaponifiable matters the following quick process is excellent, giving results within 1 per cent of the correct amount: In a flask marked at 50 cc., 100 cc., and 130 cc., saponify 2 to 3 grams of the mixture (or less weight if percentage of unsaponifiable matter is very large); fill to 50 cc. mark with alcohol, then to the 100 cc. mark with water, and to the 130 cc. mark with petroleum ether. Shake, let settle, pipette off 20 cc. of the petrolic ether solution, evaporate and weigh dry residue.

Rev. internat. falsificat., **7,** 84, 85. F.

3. Free acids.

486. Besana, C.—*Estimation of free acids in butter.*

Treatment with alcohol at 50° dissolves the free acids, which may then be titrated with $\frac{N}{10}$ sodium hydroxid, using phenolphthalein as indicator. Rancid butter does not always contain the largest proportion of free acids.

Chem. Ztg., **15,** 410; Jour. Chem. Soc., **62,** 924. J. W. F.

487. von Klecki, V., 1894.—*Investigations on the rancidity and the acid number of butter* (*Untersuchungen über das Ranzigwerden und die Säurezahl der Butter*).

Leipsic, 1894, pp. 66; Milch Ztg., **23,** 186, 187, 203, 204; Exp. Sta. Record, **5,** 1023, 1024. J. P. S.

4. Insoluble and fixed fatty acids.

488. Cassal, C. E.—*The estimation of insoluble fatty acids.*

Author employs a separating flask provided with a tapped tube with a narrow bore, for washing the insoluble fatty acids; the washing being completed, the last wash-water is run off and the insoluble fatty acids are received directly into a weighed vessel, preferably a deep and light platinum basin. The portions adhering to the sides of the apparatus are washed down with ether.

Analyst, **18,** 44, 45; Jour. Chem. Soc., **64,** ii, 308. F. W. W.

489. Schmidt and Portheil.—*Fixed fatty acids in butter.*

The method proposed is as follows: 5 grams of dry butter, 2 cc. of 50 per cent NaOH and 20 cc. of glycerol are heated in a flask of 300 cc. capacity until the mixture boils and all the water is driven off. A clear soap solution is obtained in twenty minutes; this is cooled, mixed with 90 cc. of water, which should give a very limpid solution, 50 cc. of 25 per cent sulphuric acid and a little pumice are added, and the liquid distilled until the volume is reduced to 100 cc.; the residue is then filtered through a dry filter and titrated with $\frac{N}{10}$ KOH, using phenolphthalein as indicator.

Pharm. Jour.; Rev. internat. falsificat., **6,** 49. W. F.

5. Oleic acid—precipitation method.

490. Hehner, O.—*On the determination of oleic acid.*

Author concludes from his experiments that "the method of olein determination, which is founded upon the relative solubility of the lead salts in ether, is utterly untrustworthy. Neither are the lead salts of the saturated fatty acids sufficiently insoluble, nor the unsaturated lead salts so easily soluble as to allow of a separation useful for analytical work." The experiments were made with pure butter fat, oleomargarin, almond oil, and cocoanut oil. The results obtained correspond to those found by Lewkowitsch (Jour. Soc. Chem. Ind., 1890, 845).

Analyst, **17,** 181–183. F. W. W.

6. Oleic acid—iodin method.

491. Welmans, P.—*Iodin absorption method* (*Zur Ausführung der Jodadditionsmethode*).

The author dissolves 25 grams iodin and 30 grams corrosive sublimate in sufficient sulphuric ether (or acetic ether) to make 500 cc. of the solution, and makes the volume up to 1 liter with pure acetic acid. He finds this solution much more stable than the one in common use, and it is not necessary to dissolve the fat in chloroform before the absorption of the iodin. The temperature at which the solutions are measured must be approximately the same as that at which the excess of iodin is titrated, as the coefficient of expansion of the iodin solution is quite different from that of the thiosulphate solution. The best temperature for the absorption is 17° to 20°.

Pharm. Ztg., **38,** 219; Ztschr. anal. Chem., 1893, **32,** 497; Chem. Ztg., **17,** Rep., 111. W. D. B.

492. Fahrion, W.—*Hübl's iodin numbers* (*Zur Hübl'schen Jodadditionsmethode*).

About 0.15 gram of the fat is dissolved in 10 cc. of pure chloroform, and mixed first with 10 cc. of a 6 per cent alcoholic solution of mercuric chlorid, and then with 10 cc. of a 5 per cent alcoholic solution of iodin. After two hours 20 cc. of a 10 per cent solution of potassium iodid is added, the liquid diluted to 150 cc. with water, and titrated with $\frac{N}{10}$ sodium thiosulphate, without starch. Blanks must, of course, be made.

Chem. Ztg., **16,** 862, 863; Jour. Chem. Soc., **64,** ii, 103; Ber. chem. Ges., **25,** Ref. 694. J. W. F.

493. Fahrion, W., October, 1893.—*Hübl's iodin addition method* (*Zur Hübl'schen Jodadditionsmethode*).

Reference is made to the recently reported experiments of Dieterich, Holde, and Benedikt with regard to the execution of the iodin absorption method. Their conclusions are to the effect that the direct employment of the mercuric chlorid and iodin solutions is not to be recommended, but they should be mixed at least twenty-four hours before use. The author, however, does not consider this advisable always, as tests with castor oil gave results indicating the lack of utility of the method of employing separate solutions. It is recommended that a blank test be executed at the same time and under the same conditions as the actual test, this being all the more necessary on account of the difficulty experienced in obtaining chloroform of a sufficient degree of purity.

Chem. Ztg., **16,** 1471. B. B. R.

494. Fahrion, W., March, 1893.—*Analysis of liquid fats* (*Beiträge zur Analyse der flüssigen Fette*).

A series of determinations of the iodin numbers in oils is reported, results being given as obtained both by the usual method and that devised by the author. In the latter process, the fat was completely saponified with alcoholic soda solution and the dilute alcoholic liquid neutralized exactly with HCl, using phenolphthalein as an indicator. In this solution the iodin number was determined (after two hours' standing) without addition of chloroform. In every instance the figures obtained by this method were higher than those secured by the usual process. In oils with higher iodin numbers, the method of Hehner for nonvolatile acids is not to be recommended, but the author gives a method which he claims is thoroughly reliable. This process consists in the saponification of the fat with alcoholic soda solution, expulsion of the alcohol solution in water, and addition of HCl in slight excess, followed by 100 cc. petroleum ether.

The oxy-fat acids undissolved by the petroleum ether are dissolved in warm alcohol and evaporated to dryness.

Chem. Ztg., **17,** 434. B. B. R.

495. Dieterich, E.—*Hübl's iodin addition method* (*Zur Hübl'schen Jodadditionsmethode*).

The author notes that when, in iodin absorption tests, the titre of the iodin solution is taken at the commencement of the twenty-four hours during which the absorption is to take place, the iodin numbers secured are invariably high. The iodin number is greater the higher the temperature, the older the solution, the greater the excess of iodin, and the longer the period of action of the solution. When the titre of the solution, however, is taken at the end of the experiment, the iodin number is found to be low. As the iodin titre of Hübl's solution shows a certain rate of decrease on standing, a similar allowance should be made for the decrease of the titre of the iodin excess in experiments with oils.

Helfenberger Annalen, 1891; Chem. Ztg. Rep., **16,** 243; Analyst, **17,** 219, 220.

496. Holde, D., August, 1892.—*Hübl's iodin addition process* (*Die Hübl'sche Jodadditionsmethode*).

The result of some iodin absorption tests are reported, in which a titre of the iodin solution was taken both at the commencement and end of the two hours absorption test. The results calculated from the titre of the iodin solution, made at the commencement of the experiments, gave an apparent iodin absorption of from 3.5 to 4 per cent higher than the actual absorption, employing in the calculation the value of the final titre of the iodin solution. The proposal of Fahrion to preserve the iodin and the mercuric chlorid solutions separately is to be commended, but it is advisable to mix the solutions two days before use on account of the rapid initial decline of the titre of the iodin solution. The best results were secured by employing a large excess of iodin in the absorption experiments.

Chem. Ztg., **16**, 1176–1178; Analyst, **17**, 199, 200. B. B. R.

497. Gantter, F.—*Iodin absorption (Eine neue Methode für Bestimmung der Jodzahl in Fetten und Oelen).*

The author shows that in conducting Hübl's process it is necessary that the conditions be kept absolutely constant, as varying amounts of mercuric chlorid or iodin give widely divergent results. He recommends the employment of a solution of 10 grams of iodin in carbon tetrachlorid, and dissolving the fat directly in the iodin solution. Of drying oils, 0.1 gram, of other fats, 0.2 gram is weighed into a stoppered bottle; 50 cc. of iodin solution are added, and the mixture shaken until the oil is dissolved. Sufficient water is added to form a layer several millimeters thick on the surface, the stopper inserted, and the whole allowed to react at the ordinary temperature for fifty hours. The residual iodin is then titrated by a solution of sodium thiosulphate containing 19.528 grams per liter.

Ztschr. anal. Chem., **32**, 178–184; Jour. Chem. Soc., **64**, ii, 309.

J. W. F.

F.—Volatile fatty acids.

498. Smetham.—*Rapid saponification method (Saponification rapide dans les recherches analytiques).*

Smetham claims very quick results by use of 20 cc. of ether in saponification of 3 grams of fat with alcoholic potash in a flask with a long, open tube projecting vertically. The ether is driven off with the alcohol, of course.

Bollettino Chimico Farmaceutico, **32**, 641; Rev. internat. falsificat., **7**, 63. F.

1. *Reichert-Meissl process.*

499. Richmond, H. Droop.—*The Reichert process and its modifications.*

An abstract of all papers published on the Reichert process, and the results found and conclusions drawn by the various chemists.

Analyst, **17**, 171–176. F. W. W.

500. Richmond, H. Droop.—*Notes upon the theory and practice of Reichert's process.*

Analyst, **18**, 64; Ber. chem. Ges., **26**, Ref. 390.

501. Richmond, H. Droop.—*Volatile fatty acids of butter.—Observations upon a recent contribution by W. Johnstone.*

Chem. News, **66**, 235; Ber. chem. Ges., **26**, Ref., 389.

502. Richmond, H. Droop, October, 1892.—*The Reichert process for butter, etc.*

Reference is made to the observation of Wilson (Chem. News, **66**, 199) with regard to the rate of the distillation of volatile fatty acids, the author being of the opinion that the writer referred to has arrived at his conclusions by reasoning from analogy between butyric and acetic acids. The results of Mansfield with employment of caustic potash, show a close agreement with those obtained by Wollny's method, and others have shown that the agreement is within the limits of permissible error. The author has previously shown the rate of distillation of butyric acid, and has found that when distilling 50 cc. out of 75, 89.1 per cent should pass over, and when 110 cc. out of 140 are distilled, 96.3 per cent should pass over. With Leffman and Beam's modification, employing glycerol in place of alcohol, there is very slight increase in the amount of acid distilled over. The author believes that a reasonable variation of the amount of salts or other substances tending to raise the boiling point will not affect the results within the limits of experimental error.

Chem. News, **66**, 251. B. B. R.

503. WILSON, J. A., October, 1892.—*The Reichert process for butter and other fats.*

The author calls attention to the fact observed by himself long before, that in the Reichert process the rate of distillation of volatile fatty acids increases with the proportion of salts in solution. It is therefore very necessary that in such determinations, the same quantity of acids and alkalis must be employed, if it is expected to secure an agreement in the results by different operators. If sodium hydrate be substituted for potassium hydrate (as in the Wollny process), or if oxalic or phosphoric acid be used in place of sulphuric acid, there will also be a lack of agreement in the figures secured.

Chem. News, **66**, 199. B. B. R.

504. JOHNSTONE, W., October, 1892.—*The volatile fatty acids of butter.*

In making an investigation with regard to the composition of butter fat, a sample of genuine butter showing 85.6 per cent of insoluble fatty acids was subjected to examination. In determining the volatile fatty acids by the Reichert-Wollny process, it was found that solid volatile fatty acids collected in the tube of the condenser to the amount of 9.31 per cent of the total weight of the butter. A quantitative examination of this deposit showed that it consisted of 18.6 per cent pseudo-oleic acid and 81.4 per cent myristic acid.

Chem. News, **66**, 188; Ber. chem. Ges., **26**, Ref., 389. B. B. R.

505. BÜNTE, C., 1894.—*Concerning the suggested modifications of the Reichert-Meissl butter test for margarin, and a new method for the exact determination of the Reichert-Meissl number (Ueber die in Vorschlag gebrachte Modification der Reichert-Meissl'schen Butterprüfung auf Margarinzusatz und eine neue Methode zur exacten Ermittlung der Reichert-Meissl'schen Zahl).*

The author had been unable to obtain sufficiently accurate results by the old Reichert-Meissl method, and suggests the following modification as being both practicable and accurate.

Place an Erlenmeyer flask of 1 liter capacity containing about 5 grams of the weighed butter fat in a drying bath until the temperature reaches 100° C.; then add 10 cc. of sulphuric acid (sp. gr. 1.8355). During this time the flask should be shaken and the fat brought into solution. The flask is then put on a water bath at 30° to 32° C., for ten minutes, and 150 cc. of water added, the flask being shaken continuously; titre with concentrated permanganate solution, shaking the flask until the red color is held for a moment; and proceed as in the old method.

The author has found by experiment that all the other modifications hitherto proposed are either scientifically unsound or useless in practice. He also holds that in order to secure results as free from error as possible, it is necessary to consider the limitations as to temperature, concentration of the acid, and size of flask, to which he has called attention in his article.

Chem. Ztg., **18**, 204, 206; Chem. Centralbl., 1894, i, 527; Ber. chem. Ges., **27**, Ref. 143; Jour. Soc. Chem. Ind., **13**, 427. J. P. S.

2. *Leffmann-Beam method.*

506. LEFFMANN, H., and BEAM, W.—*The alkali-glycerol as the saponifying agent in the Reichert process.*

Note supplementing the description of the process in Analyst, August, 1891; the previous evaporation of water from the alkali-glycerol may advantageously be omitted.

Analyst, **17**, 65, 66. F. W. W.

507. **Partheil, A.**—*Estimation of the volatile fatty acids in butter fat* (*Ueber die Bestimmung der flüchtigen Fettsäuren des Butterfettes*). (*Leffmann-Beam method.*)

Five grams of filtered, dry butter fat is placed in a flask of 300 to 350 cc. capacity and 2 cc. of 50 per cent aqueous soda, and 20 cc. of glycerol added. The mixture is then carefully boiled until all the water is expelled, the flask being gently rotated and the heating continued with a smaller flame. After fifteen to twenty minutes a clear soap solution is obtained, which, after cooling, is mixed with 90 cc. of water and 50 cc. of sulphuric acid (50 cc. of pure acid to the liter), some fragments of pumice added, and 110 cc. distilled over and titrated. The method is more convenient and quicker, but the test analyses are not entirely satisfactory.

Apoth.-Ztg., **7,** 435; Chem. Centralbl., 1892, ii, 635, 636; Jour. Chem. Soc., **64,** ii, 197. J. W. F.

3. König's modification.

508. **Kreis, H.,** and **Baldeis, W.**—*Determination of volatile fatty acids in butter.*

Authors studied König's modification of the Reichert-Meissl method, in which a determined quantity of alcoholic barium hydrate is added to the distillate, and the free baryta determined by back titration. The errors were found to be the same as in the unmodified process. Saponification by sodium alcoholate (as proposed by Kossel and Obermüller) was found to give more complete and rapid saponification.

Chem. Centralbl., 1892, i, 1005; Centralbl. Agrik. Chem., **22,** 210. W. F.

4. Kreis's modification.

509. **Kreis, H.,** September, 1892.—*Modification of the Reichert-Meissl method* (*Modification der Reichert-Meissl'schen Methode der Butterprüfung auf Margarin*).

The author claims that butter fat and margarin fats, at the ordinary temperature, can be dissolved in concentrated sulphuric acid with great ease, the glycerol and the fatty acids being completely separated without destruction of the decomposition products. By means of a large number of experiments the conditions were determined, by observance of which the evolution of sulphurous acid was reduced to a minimum and results agreeing with the Reichert-Meissl process were obtained. Five grams of fat are brought into an Erlenmeyer flask of 1 liter capacity, heated to melting, and mixed with 10 cc. H_2SO_4 of 1.825 specific gravity. After agitation for a short time, the contents of the flask become perfectly clear; the flask is now kept for ten minutes in a water bath at 30° to 32° C., 150 cc. water are added, with thorough shaking, and the distillation performed as usual. With butters the results agree quite well with those of the Reichert-Meissl process, but with margarin, and with mixtures containing margarin, the figures are considerably higher. This latter fact is considered as affording a means of detecting mixtures of margarin with butter. Margarin does not, moreover, become clear near so quickly as butter after treatment with sulphuric acid.

Chem. Ztg., **16,** 1394. B. B. R.

510. **Pinette, J.**—*A modification of the Reichert-Meissl method* (*Eine Modification der Reichert-Meissl'schen Methode der Butterprüfung auf Margarin*).

Reference is made to the method proposed by Kreis for the determination of volatile acids and its application to testing for margarin in butter. The chief objection to the process is that considerable quantities of sulphurous acid are formed which, in a number of experiments, caused enormously high figures to be secured. The distillate, after titration, however, can be oxidized by means of bromin water, and, after determining the amount of sulphuric acid formed, its equivalent can be deducted from the figures previously obtained, though, even in this way, erroneous results were not entirely avoided. The following modification of Kreis' method is proposed: After treating the butter fat with sulphuric acid, and completion of the solution, 150 cc. of water are added and a strong solution of potassium permanganate is run in until the red tint is permanent, even after shaking, for several seconds. The odor of sulphurous acid is no longer perceptible and the solution may be distilled at once. This modified method is more expeditious than the Reichert-Meissl, and, in conjunction with the latter process, the detection of margarin in butter can be effected.

Chem. Ztg., **17**, 395; Chem. Centralbl., 1893, i, 803; Rev. internat. falsificat., **6**, 157; Analyst, **18**, 145. B. B. R.

511. MICKO, K.—*Kreis' modification of the Reichert-Meissl method* (*Zur Butterprüfung auf Margarin*).

The author notices the generation of sulphurous acid which affects the titration of the fatty acids, and proposes to oxidize the sulphurous acid with potassium bichromate. Four cubic centimeters of 4 per cent bichromate of potash is added just before distilling, and, after shaking, a slight excess of sulphate of iron is added. This method is said to give figures agreeing well with the Reichert-Meissl method.

Ztschr. allgem. Oesterr. Apotheker-Ver., **31**, 229; Chem. Centralbl., 1893, i, 803, 804; Chem. Ztg. Rep., **17**, 113; Rev. internat. falsificat., **6**, 157; Ztschr. Nahr. Untersuch., **7**, 37, 38. E. W. A.

512. PRAGER, A., and STERN, J., April, 1893.—*Kreis' modification of the Reichert-Meissl process* (*Zur Kreis'schen Modification der Reichert-Meissl'schen Butterprüfung auf Margarin*).

Tests made by the authors confirm the results already reported by Pinette and Micko, the figures obtained by Kreis' method being in every case higher than the Reichert-Meissl numbers. The removal of the sulphurous acid formed in using Kreis' process can be effected by conducting a stream of air through the sulphuric acid solution of the fat, this being followed by a current of carbon dioxid, and finally by still another current of air. The fat is treated with sulphuric acid as in Kreis' method, being allowed, however, to cool for ten minutes, with continuous agitation. Air is now conducted through the liquid until the odor of sulphurous acid is no longer perceptible; 100 cc. of water are added cautiously, with shaking, keeping the liquid as cool as possible. Carbon dioxid is now passed through the solution for ten minutes, followed by a stream of air for another ten minutes. The distillation is now effected in the usual manner. The figures reported show a comparatively close agreement with those of the Reichert-Meissl process.

Chem. Ztg., **17**, 468; Chem. Centralbl., 1893, i, 861; Analyst, **18**, 145. B. B. R.

513. PRAGER, A., and STERN, J., June, 1893.—*Saponification of fats by Kreis' process* (*Ueber die Verseifung von Fetten nach Kreis und deren Modificationen*).

The authors refer to a recent article of Kreis in which he prescribes in detail the manner of testing the acid employed, and state that by observing the proper precautions figures can be secured approximating the Reichert-Meissl numbers very closely. The statement of Kreis that relatively higher figures are secured for margarin and mixtures of margarin with butter than by the Reichert-Meissl process is also confirmed, the results of a number of analyses being given to substantiate this fact. This process is, therefore, considered a valuable aid to the detection of margarin in butter, the presence of 10 per cent of margarin being readily perceptible in the increase of the figures secured on distillation as compared with those obtained by the usual method. The results secured by the method of Pinette are much lower than those of the Reichert-Meissl process, while the modification proposed by the authors themselves yields figures which agree quite well with those resulting from the use of the ordinarily employed method.

Chem. Ztg., **17**, 880. B. B. R.

514. SCHATZMANN and KREIS, April, 1893.—*The saponification of fats with sulphuric acid* (*Ueber die Verseifung von Fetten mit conc. Schwefelsäure*).

Reference is made to Pinette's proposed modification of Kreis's method and to his statement that it was impossible to secure correct results by the employment of the original process. The authors fail to see why better results were not obtained by Pinette, unless it were possible that in the original description of the method sufficient emphasis was not laid upon the degree of concentration of the acid employed. The proper strength can not readily be arrived at by means of the areometer, but is determined by actual tests with butter and with oleomargarine; the former should be dissolved clear at once and the latter after two to three minutes. The sulphuric acid of commerce is in most cases too strong, the acid employed by the authors containing only 91.53 per cent H_2SO_4. By employing this acid and observing proper precautions, this method gives results agreeing quite well with the Reichert-Meissl process. The modifications of Pinette, and of Prager and Stern are considered entirely superfluous. A series of determinations made by former process yielded entirely too low results.

Chem. Ztg., **17**, 544. B. B. R.

5.—Schmidt's modification.

515. SCHMIDT, August, 1892.—*Analysis of butter* (*Ueber Butter-Untersuchungen*).

The use of Reichert's method is somewhat objectionable on account of the employment of alcohol, which is difficult of expulsion and gives rise to the production of ethers, the accuracy of the results being perceptibly affected.

The method proposed avoids the employment of alcohol, 5 grams of butter being placed in a flask along with 2 cc. strong soda solution (1:1) and 20 cc. glycerol. After ten minutes careful heating the saponification is completed, and the usual process is followed after addition of dilute sulphuric acid.

Chem. Ztg., **16**, 1275. B. B. R.

G.—RANGE IN VOLATILE FATTY ACID PERCENTAGES.

516. KREIS, H.—*Butter-testing.*

The author, from the results of a large number of experiments, confirms the statement of Schrodt and Henzold to the effect that the Reichert-Meissl number for butters is dependent upon the period of lactation. During the year 75 samples which were tested showed results ranging from 21.1 to 34.4. The highest figures were secured during the earlier stages of lactation.

Schw. Woch. Chem. Pharm., **30**, 449; Chem. Ztg. Rep., **16**, 351. B. B. R.

517. VIETH, P.—*Low volatile fatty acids in pure butter.*

Analyses of 209 London market butters often gave by the Reichert-Wollny method a coefficient of less than 25, in terms of $\frac{N}{10}$ alkali. Such low results were repeatedly observed in butters known to be pure, and once in the case of a month's product from a certain dairy.

Milch-Ztg., 1892, 330 and 352; Centralbl. Agrik. Chem., **22**, 286. W. F.

518. MAYER, ADOLPHE.—*Variation of volatile fatty acid in pure butter*

Rev. internat. falsificat., **6**, 69, 70. W. F.

H.—ADULTERATION TESTS.

519. WANTERS.—*Analysis of butter* (*L'analyse du beurre*).

The author reports the results of the examination for adulterations of a large number of samples of butters and compares the merits of the different processes employed for the detection of adulterants. Butters giving a sp. gr. below 0.865 were classed as adulterated, as well as those samples (179 out of 508) showing over 90 per cent fixed acids. Butters giving refractive tests below 26° were considered suspicious. The pure butters of known origin, by the Reichert-Meissl process, gave figures varying from 34.7 to 28.7. The author concludes that no single test can be absolutely relied on in the detection of adulterations, on account of the very considerable variations in certain particulars exhibited by perfectly pure butters.

Bull. Assoc. Chim. Belge., **6**, 178. B. B. R.

520. JOHNSTONE, W.—*Recognition of foreign fats in butter.*

The author claims to have found that pure butter does not contain triolein and tributyrin, but a fat which he designates as "oleovacciobutyrat." His method depends upon the fact that the relation between the oleic and butyric acids in this fat is constant. An excess of oleic acid indicates the addition of an animal fat, and an excess of butyric acid indicates an admixture of vegetable oils. From the amount of butyric acid found in a sample of butter the oleovacciobutyrat is calculated by means of the factor $\frac{650}{88}$. The iodine number of the butter is determined and calculated to the proper glyceride by multiplying by $\frac{10}{9} \times \frac{650}{282}$. The butter is not pure unless the results of the two calculations agree. It is claimed that with this method the addition of 3 per cent of foreign fat to butter can be recognized. The method is covered by an English patent.

Chem. Centralbl., 1893, I, 631. E. W. A.

521. KÖNYÖKI, A.—*Adulteration of butter.*

Ztschr. Nahr. Untersuch., **7**, 53, 54; Chem. Centralbl., 1893, i, 804.

522. HEHNER, O.—*Notes on butter.*

Finds no definite distinction between pure and adulterated butters as to manner of melting, and cautions analysts against placing too much reliance upon any test based on this principle.

Analyst, **17**, 102. F. W. W.

523. ZUNE, March, 1893.—*Analysis of butter* (*L'analyse des beurres*).

Reference is made to the reaction proposed by Jean for the detection of cocoa fat in butter by heating the sample in the presence of sulphuric acid and alcohol. This treatment gives rise to the formation of an ether (coccinic (?)) whose very characteristic odor permits the detection, it is claimed, of as little as 10 per cent cocoa fat in butter. This test, the author states, is not conclusive as to the presence of cocoa fat, since the same reaction is noted where lauric and myristic acids are present. The author also notes that the insoluble fat acids of cocoa fat lose continually in weight when dried at 100° C. (or an even higher temperature), so that a constant weight is not readily attainable in the presence of considerable proportions of this substance.

Bull. Assoc. Chim. Belge., **6**, 249. B. B. R.

524. CAVALLI, A.—*Detection of foreign fats in butter* (*Falsification du beurre par les graisses étrangères*).

Author says that 4 grams of butter, not too rancid, filtered and mixed with 0.5 grams of copper turnings and 1 cc. of nitric acid (sp. gr. 1.42), and heated on a water bath will give with pure butter a yellowish green tint, deepening with time. Pure oleo is yellowish at first, then more or less yellow; after forty-five minutes reddish, and after one and one-half hours reddish. A mixture of butter and oleo behaves like oleo, and a sharp reaction is given by a mixture containing 20 per cent of the latter. A mixture of butter and lard gives a coloration reddish at first, and more intense in thirty minutes; an addition of 10 per cent of lard can be detected.

Rev. internat. falsificat., **7**, 61. F.

525. HAIRS, EUG.—*Modification of Maumenes test applied to butter and oleo oil* (*margarin*) (*Sur l'échauffement sulfurique du beurre et de la margarine*).

Run into a 60 cc. matrass 5 cc. of strong H_2SO_4 (sp. gr. 1.843). Weigh on a balance sensible to 1 cgm. Run slowly onto the surface of the acid 2.5 grams of melted, filtered butter, not above 30° C. in temperature. Close the matrass with a rubber stopper, through which passes a thermometer whose bulb is immersed partly in the fat, partly in the acid. Put into a water bath at 33° C., and wait till the thermometer marks 30° C. Lift the matrass from the bath, wrap bulb in a towel to avoid loss of heat, shake vertically twelve to fifteen times, so as to mix the two layers, holding the stopper in firmly. Read the thermometer as soon as it stops rising. Subtract 30° C. from the reading to get the elevation of temperature due to the reaction. Ten samples of butter thus examined gave elevation of 24° to 28.7°; 11 of margarin from 30.9° to 49.3°. But various mixtures of the two gave elevations ranging from 27.3° to 32°. Evidently the test is not infallible for the detection of adulterations.

Jour. Soc. Pharm. d'Anvers; Rev. internat. falsificat., **6**, 192, 193. F.

526. RODEWALD, H.—*Detection of margarin in butter.*

Butter and margarin differ only in their quantitative composition, their qualitative reactions, volatile and insoluble fatty acids, combination with iodin, and refractive property being common to both. Comparison of the numbers obtained with the minimum and maximum numbers for natural butter is unsafe, inasmuch as other maxima and minima may be found. Still more difficult is the case when the numbers for the suspected sample fall between the minimum and maximum numbers for natural butters. The object of the present paper is to indicate how, in such cases, the probability of adulteration may be arithmetically established from all the results. Schrodt and Henzold's numbers are employed.

Landw. Vers.-Stat., **40**, 265–275; Jour. Chem. Soc., **62**, 1034; Exp. Sta. Record, **4**, 96. J. W. F.

527. JEAN, F.—*The acetic acid test for fats.*

The acetic acid used is glacial acetic acid, to which enough water has been added to raise the density to 1.0565 at 15° C. Three cubic centimeters of oil or fat are introduced into a tube 1 cm. in diameter, and graduated in one-tenth of a cubic centimeter; the tube is placed in a water bath at 50° C., and the volume of fat accurately adjusted to 3 cc. Three cubic centimeters of acetic acid, measured at 22° C. are then poured in from a pipette. The fat and the acetic acid are shaken several times to insure complete admixture, and the layers allowed to separate completely in the bath at 50° C. By subtracting the quantity of acetic acid from 3, and multiplying by 33.3, the percentage of acetic acid dissolved out by the fat is obtained. Numerous samples of butter were found to dissolve 63.33 per cent acetic acid; 4 butters from Touraine, however, dissolved 73 per cent; margarin, 26.7 to 40 per cent; tallow, 26.7 per cent. Author concludes that this method, with the oleorefractometer and the R.-W. method, will establish adulteration in butter.

Ind. lait., 1892, 205; Analyst, **17**, 194, 195. F. W. W.

528. HOUZEAU, A.—*Preliminary note on a new method of detecting the adulteration of butter (Recherches pour établér les bases d'une nouvelle méthode destinée à reconnaître la falsification des beurres, etc.).*

The method depends (1) on the less solubility of adulterated butter in alcohol of given strength, at given temperature, and in given proportion, as compared with pure butter; and (2) on the fact that while pure butters do not all become cloudy in solutions otherwise similar, at precisely the same temperature, a twice-repeated solution and recovery of the fats soluble in alcohol of given strength results in the separation of fats that do cloud at the same temperature, other conditions equal; but that adulterated butters thus treated still tend to require a higher temperature if adulterated with animal fats, or a lower if with vegetable fats. A proper proportion of the two will, it is true, give a mixture behaving like butter, but a parallel test with the Reichert-Meissl process promptly reveals the fraud. These principles are not yet worked out into a method.

Compt. rend., **116**, 952; Rev. internat. falsificat., **6**, 171, 172; Chem. Centralbl., 1893, i, 1094. W. F.

529. ERDÉLYI, J.—*Detection of foreign fats in butter.*

Pure butter fat dissolved in three times its volume of cumene (boiling point, 165°C. at 758 mm.) gives a solution which remains clear at 0° C. for at least an hour, while mixtures of butter with margarin or with lard become turbid much earlier. Filter the fat, cool it for twenty-four to forty-eight hours in a refrigerator, place 2 cc. of it with 6 cc. of cumene in a clear, dry test tube of 2 cm. diameter, and after twenty-four hours at the ordinary temperature, plunge the tube into powdered ice, and after the lapse of an hour examine it at short intervals.

Ztschr. anal. Chem., **31**, 407–410; Jour. Chem. Soc., **62**, 1532; Ber. chem. Ges., **25**, Rep., 875; Chem. Centralbl., 1892, ii, 761. J. W. F.

530. Brullé, Raoul, July 15, 1893.—*Method of detecting adulteration of butter* (*Méthode générale pour l'analyse des beurres*).

1. The presence of seed oils can be detected by heating 12 cc. of the butter in a test tube with 5 cc. of a 2.5 per cent silver nitrate solution. The coloration changes are very characteristic, and the microscopic form of the crystals deposited on cooling will reveal the presence of olive oil, which gives no color reaction.

2. To detect animal fats: Melt and filter the butter. Heat 6 cc. in a flat dish, 7 cm. in diameter, on an oil bath at 148° C. When the butter attains a temperature of 130° add a pinch of powdered pumice and 8 drops of fuming nitric acid. Mix and heat about twelve minutes, then cool quickly to 21° C. After an hour, determine the penetrability of the fat by a sharp rod surmounted by a flat plate on which weights may be placed and sliding in a holder. Weights are added until the end of the rod sinks suddenly into the fat. With a pure butter, 250 grams sufficed; with a pure margarin, 5,000 grams were required. The intermediate figures permit valuation by interpolation. In the additional presence of plant oils, the oleogrammeter results are affected, but still answer the purpose. Over 200 tests at the laboratory of the Société des Agriculteurs de France upon known mixtures, under the supervision of a commission, always gave results in conformity with the truth.

Jour. Pharm. Chim. [5], **28**; Jour. Soc. Chem. Ind., **12**, 717; Rev. internat. falsificat., **6**, 212–213.; Exp. Sta. Record, **5**, 450; Compt. rend., **116**, 1255–1257; Ber. chem. Ges., **26**, R, 503. F.

531. Mariani, G.—*The detection of oleomargarine in butter* (*Ueber das Brulle'sche Reagens, um Oleomargarin in der Butter zu erkennen*).

The reagent proposed by Brullé (a solution of 25 parts silver nitrate in 1,000 parts of 95 per cent alcohol) was tested to ascertain its utility for detecting the presence of oleomargarine in butter. The reagent was not reduced by pure fresh butter, fresh oleomargarine, nor by mixtures of the two, but when these substances are rancid the reduction, with formation of the brown color, takes place very readily. Lard also has a reducing effect upon Brullé's solution. The reagent is pronounced of no utility in testing for oleomargarine in butter.

Selmi, **3**, 38; Chem. Ztg. Rep., **17**, 55. B. B. R.

532. Gantter, F.—*Discrimination of butter from margarin.*

The author's method for the estimation of cotton-seed oil in lard has been found to work equally well for the detection of margarin in butter. Genuine butter gives only a straw-yellow to reddish-yellow color with sulphuric acid, while most of the oils used in making margarin give a dark brown color.

Ztschr. anal. Chem., **32**, 411–413; Jour. Chem. Soc., **64**, A, 603. J. W. F.

533. Seyda and Woy, 1894.—*Comparison of the ordinary chemical methods for the testing of butter adulterated with margarin* (*Vergleichung der bei Prüfung von Butter auf Margarinzusatz üblichen chemisch-analytischen Methoden*).

Chem. Ztg., **18**, 906, 907. J. P. S.

534. Houzeau, A.—*A new method for testing mixtures of butter and foreign fats.*

Compt. rend., **116**, 1100–1103; Ber. chem. Ges., **26**, Ref. 503.

535. Weilandt, M.—*Behavior of butter and margarin toward coloring matters.*

Milch-Ztg., 1892, 238–241; Exp. Sta. Record, **4**, 97. E. W. A.

I.—MISCELLANEOUS.

536. PIZZI, A.—*Chemical examination of butter fats.*

Staz. Agr. Ital., **25**, 101–118; Chem. Centralbl., 1894, i, 142.

537. SOHN, C. E.—*Simple apparatus for the estimation of the consistency of semisolid substances, with a notice of a new method of testing butter.*

Analyst, **18**, 218; Chem. Ztg., **17**, Repert., 251; Jour. Soc. Chem. Ind., **12**, 866.

538. GARELLI, F., and CARCANO, L.—*The use of the Raoult kryoscopic method in the examination of butter.*

Staz. sper. agr. Ital., 1893, Aug. 24; Milch-Ztg., **23**, 52.

539. LAVES, E., 1893.—*The use of barium hydroxid in the analysis of butter* (*Ueber die Verwendung von Barym-hydroxyd in der Butteranalyse*).

Arch. Pharm., **231**, 356–366; Ber. chem. Ges., **26**, Ref., 727; Chem. Centralbl., 1893, ii, 298. J. P. S.

540. WRAMPELMEYER, E.—*The lecithin content of butter.*

Landw. Vers.-Stat., **43**, 437, 438; Chem. Ztg., **17**, Repert., 260; Ber. chem. Ges., **27**, Ref. 143; Exp. Sta. Record, **5**, 342.

CHEESE.

541. MAGGIORA, ARNOLDO.—*Analysis of cheese* (*Analyse du fromage*).

The author recommends the following procedure: Beat one-third or one-fourth of a slice in a mortar to a paste, press through a sieve of less than 1 mm. mesh, mix carefully and keep in a stoppered flask. Estimate moisture in 2 to 3 grams, mixed with known weight of quartz sand and dried to constant weight at 100° C. Estimate fat by extraction of the cheese thus dried, by petroleum ether (B. P. 45°) in a Soxhlet apparatus. Estimate crude protein by multiplying by 6.25 the percentage of nitrogen determined by the Kjeldahl method. Determine pure albuminoids by Stutzer method, multiplying by 6.25 as before, and using 2 grams of the sample. To estimate ammoniacal nitrogen work 2 to 3 grams up into an emulsion with distilled water in a mortar, adding the water gradually until there is about 250 cc.; treat with 5 grams of calcined magnesia and distil. The amids are computed by difference. Estimate ash in 5 grams by burning, taking up with boiling water, and completing combustion of the carbon. After weighing ashes dissolve in nitric acid, determine chlorin gravimetrically, calculate it as salt, and deduct from total ash.

Rev. Internat. Falsificat., **7**, 46; Chem. Centralbl., 1894, i, 118. F.

542. SPICA and DE BLASI.—*Chemical researches on Sicilian cheese.*

Staz. sper. agr. Ital., **23**, 133; Analyst, **18**, 94–96. F. W. W.

543. BONDZYNSKI, STEFAN.—*Fat determination in cheese.*

From a weighing tube weigh off some fixed quantity of finely divided cheese into a tube blown to a long wide bulb in the lower part, but drawn to a narrower, carefully calibrated tube at the top. Add 20 cc. of HCl, 19 per cent (specific gravity 1.1), heat carefully over wire gauze until the cheese is dissolved; the fat gathers at the surface in a melted layer. Cool the tube, add about 30 cc. of ether; this promptly dissolves the fat, even without shaking; bring to a temperature of 40° C. and let stand for a time. The acid and ether layers separate sharply, even without a centrifuge. Read off the volume of the ethereal fat solution, withdraw 20 cc. of it by means of a pipette, evaporate in a tared flask, and weigh the residual fat. Calculate to amount in total observed volume of ethereal fat solution. Excellent duplicates are obtained. The discoloration of the acid causes no error in the result.

Ztschr. anal. Chem., **33**, 186–189; Agric. Science, **8**, 137; Ber. chem. Ges., **27**, Ref. 344. F.

544. Béchamp, A.—*Studies on casein.*

Bull. Soc. Chim., **11** [3], 152–176; Exp. Sta. Record, **5**, 1008, 1009.

OTHER ANIMAL FOOD PRODUCTS.

Lard.

545. Goske, A.—*The analysis of steam lard.*

Chem. Ztg., **16** (1892), 1560, 1597; Analyst, **18**, 18–20. F. W. W.

546. Amthor, C., and Zink, J.—*Analysis of lard.*

Ztschr. Anal. Chem., **31** (1892), 534–37; Analyst, **18**, 20, 21 F. W. W.

547. Wellmans, P.—*Detection of vegetable oils in lard.*

The reaction depends on the presence of alkaloids in the vegetable oils. To 1 gram or 25 drops of lard is added 5 drops of chloroform and 2 cc. of phosphomolybdic acid solution, together with a few drops of nitric acid, the mixture being well shaken. The presence of alkaloids or glucosids causes reduction, accompanied by a green coloration. Purified oils do not react in this way. Picric acid in ethereal solution, when shaken with 10 cc. of the oil, gives a brown coloration.

Pharm. Ztg., **36**, 798, 799; Chem. Centralbl., 1892, i, 417; Jour. Chem. Soc., **62**, 1135, 1136; Analyst, **17**, 59, 60. J. W. F.

Lecithins in Eggs.

548. Maxwell, W., 1893.—*Estimation of lecithins in eggs.—Movement of the element phosphorus in the mineral, vegetable, and animal kingdoms, and the biological function of the lecithins.*

The egg materiel, boiled with alcohol and ground in a mortar, is reextracted for ten hours with ether. The alcohol and ether extracts are mixed, evaporated to dryness, mixed with alkali salts, and ignited, converting the phosphorus of the lecithins into phosphates of sodium and potassium, in which the phosphorus is determined as magnesium pyrophosphate, and the lecithin-content calculated by means of Hoppe-Seyler's factor.

Am. Chem. Jour., **15** [3], 188—(185–195). J. W. F.

Peptones.

549. Hallopeau, L. A.—*Estimation of peptone in presence of syntonin, albumen, and hemialbumose (Dosage de la peptone par precipitation à l'état de peptonate de mercure).*

Exactly neutralize liquid, presumably acid with HCl, by adding Na_2CO_3. Wash precipitate with cold water, dry at 105° C. and weigh as syntonin. Add to filtrate a drop of acetic acid, heat on water bath for thirty minutes, collect any flocky albumen precipitate, wash, dry, and weigh as usual. To second filtrate add HNO_3, drop by drop; if cloudy, stop adding acid; stir an instant, let hemialbumose settle, filter, and wash with a little water slightly acid with HNO_3. [Strictly, precipitate should be redissolved in excess of HNO_3, the solution exactly neutralized, and precipitated by saturation with NaCl, washed, dried, and weighed.] The third filtrate contains only peptone. Neutralize completely, and precipitate by excess of pure mercuric nitrate; filter, wash with cold water till free from mercury, dry at 106° to 108°; the weight of precipitate multiplied by 0.666 will give the corresponding peptone. The commercial mercuric nitrate contains free HNO_3. Its solution (1:10 or 1:7) is heated to boiling, a few drops of NaOH added until mercuric oxid formed, no longer redissolves. Cool, and make up to 1 liter.

Rev. internat. falsificat., **6**, 29–30; Compt. rend., **115**, 356–358; Bull. Soc. Chim., [3] **7**, 718–719; Jour. Chem. Soc., **64**, ii, 104. W. F.

550. Stutzer, A., September, 1892.—*Estimation of the nitrogenous constituents of commercial peptones (Zur Analyse der in Handelspeptonen vorhandenen stickstoffhaltigen Bestandtheile).*

The value of commercial peptones is influenced chiefly by the amount of albumose and peptone contained. An aqueous solution of the sample is acidified with acetic acid and heated to boiling, and after filtration the nitrogen is determined in both the residue and filtrate, soluble and insoluble albuminous substances being reckoned from the nitrogen content. Albumose, gelatin, and peptone are precipitated by means of absolute alcohol from a fresh solution, while the gelatin-peptone is precipitated from its dealcoholized solution by means of mercuric oxid paste. The peptone is separated from albumose and gelatin by insolubility of the latter in ammonium sulphate. The gelatin is determined by employment of the viscosimeter. The nitrogen in all the precipitates obtained is determined by Kjeldahl's method, and the factor 6.25 is employed in calculating results.

Ztsch. anal. Chem., **31**, 501. B. B. R.

MISCELLANEOUS ANALYTICAL PAPERS.

551. Behrens, Th.—*A system of microchemical qualitative analysis.*

Ann. de l'École polyt. Delft, **6**, 82–176; Bull. Soc. Chim. [3], **8**, 1032–1035. W. F.

552. Malfatti, H.—*An attempt to determine carbon, nitrogen, and hydrogen simultaneously.*

Ztschr. anal. Chem., **32**, 754–761; Chem. Centralbl., 1894, i, 655–656; Ber. chem. Ges., **27**, Ref. 207.

553. Klingemann, F., 1893.—*A method for the simultaneous estimation of carbon and nitrogen in organic compounds.*

Lieb. Ann., **275**, 92–102; Ber. chem. Ges., **26**, Ref. 503; Chem Ztg., **17**, Repert., 197. J. P. S.

554. Heidenhain, H., 1893.—*Determination of organic matter by moist oxidation.*

Author reviews earlier attempts to use chromic acid for this purpose, and then gives his own method. The reduction is carried out in a round-bottomed flask of 200–250 cc. capacity, into the neck of which a V-shaped glass tube with long limbs is inserted, and through which a current of cool water constantly passes. Twenty-five cubic centimeters of a solution of the organic substance, not stronger than $\frac{N}{5}$, is mixed with 30 cc. of $\frac{N}{5}$ solution of potassium bichromate, and 33 cc. concentrated, c. p., sulphuric acid are added. The whole is heated over wire gauze to boiling, and the flame then so regulated that ebullition occurs from time to time, the condenser being kept in action. After boiling them for ten minutes remove flask, and without necessarily either cooling or diluting, determine amount of unreduced chromic acid by means of solution of potassium ferricyanid and ferrous sulphate or ammonium ferrous sulphate. The above proportions are not arbitrary, but fixed as result of experiment. The method is regarded not as absolute, but solely as empirical. Complete oxidation is possible with only a few substances, being more complete the larger the excess of chromic acid; but the method, used relatively, gives results so close that if two points, as 100 per cent and 40 per cent, be fixed for a given substance, interpolation gives close enough figures. Duplicates agree well, and the method is quick. The degree of oxidation found in different cases for $\frac{N}{5}$ solution are: Oxalic acid, 100 per cent; tartaric acid, 99.48 to 99.56 per cent; dextrose, 98.45 per cent; corn starch containing 11.5 per cent of water, 85 per cent; pure glycerol, 97.5 to 97.73 per cent; acetic acid, 0 per cent; alcohol, 33.3 per cent;

a-naphthol, 75.0 per cent; *b*-naphthol, 83 per cent; benzoic and salicylic acid, little more than 90 per cent; morphine, 61 per cent.

Jour. Am. Chem. Soc., **15,** 71–77. W. F.

555. BERTHELOT, 1892.—*A new method of organic analysis* (*Sur une nouvelle méthode d'analyse organique*).

Substance is burned in a calorimeter in oxygen with a pressure of 25 atmospheres. Rapid and complete combustion is secured; the gases are then sucked out by a mercury pump and passed through the ordinary absorbents. To determine hydrogen, it is needful also to heat the calorimeter slightly to vaporize all the water formed. By adding 10 cc. of water beforehand, sulphur may be obtained in solution as H_2SO_4 and accurately determined.

For chlorin an aqueous solution of arsenious acid may be introduced instead of water; or if the substance is very rich in chlorin a small quantity of camphor may be added.

Bull. Soc. Chim. [3], **7,** 430, 431. W. F.

556. PAYNE, H. L.—*Preparation of a standard iodin solution.*

As a standard iodin solution alters on keeping for a long time, the author proposes the following quick method where a standard solution of permanganate is on hand: 10 grams of potassium iodid are dissolved in water in a liter flask and acidified with sulphuric acid. The calculated quantity of potassium permanganate is run in, and when the reaction is complete, the whole is made up to 1 liter. A clear solution of known iodin content is thus obtained, 5 atoms of iodin being liberated for each molecule of potassium permanganate.

Chem. News, **66,** 286; Jour. Chem. Soc., **64,** ii, 234. J. W. F.

557. GRÜTZNER, B.—*Stability of potassium permanganate solutions.*

A 0.1 per cent solution was unaltered at the end of a year, whether exposed to the light or kept in the dark. After one and one-half years the same solution had lost 2.61 per cent of its content of potassium permanganate when exposed to the light, and 0.94 per cent when kept in the dark. A 0.3 per cent solution was unaltered in every case at the end of one and one-half years. $\frac{N}{10}$ sodium thiosulphate was kept unchanged for six months, light having been excluded.

Arch. Pharm., **230,** 321–324; Jour. Chem. Soc., **62,** 1524. J. W. F.

558. GOOCH, F. A., and DANNER, E. W., 1892.—*On certain points in the interaction of potassium permanganate and sulphuric acid.*

Am. Jour. Sci., 1892, 301–310. J. W. F.

559. LIEBEN, A.—*Errors introduced by sulphur in coal gas.—Errors arising in chemical operations owing to the employment of gas flames.*

The author states that substances of a basic nature absorb quantities of sulphuric acid produced by the combustion of coal gas, thus causing errors, the quantity depending upon the size of the flame and the duration of the process.

Monatsh. f. Chem., **13,** 286–298; Jour. Chem. Soc., **62,** 1374. J. W. F.

560. MACFARLANE, THOMAS.—*On the use of chrysolite fiber in proximate organic analysis.*

Author describes in an exhaustive paper the various uses to which asbestos chrysolite may be put in the laboratory. Methods of analyses of milk, butter, cheese, fats and oils, soap, spirits and malt liquors, sirups and molasses, and coffee are given, as well as suggestions as to further extension of the method for other purposes.

Analyst, **18,** 73–90. F. W. W.

561. FREAR, WILLIAM, August, 1892.—*Limits of error in sampling yard manure.*

Author gave the result of experiment in which samples of equal weight were taken from each load of two different manure heaps, as it was drawn to the field.

and these samples divided, after careful mixing, until four subsamples were obtained to represent each heap. Complete duplicate analyses of each showed that, notwithstanding the careful sampling, there was nearly as great a difference between duplicate samples from the same heap as between those from the heaps differently treated. It is therefore recommended that triplicate samples be taken of such material for analysis.

Agric. Science, **6**, 475. W. F.

562. Frear and Holter.—*Loss of nitrogen in air drying.*

Agric. Science; Centralbl. Agrik. Chem., **22**, 137. W. F.

563. Wilson, J.—*Determination of sugar in soap.*

Chem. News, **64**, 28; Bull. Soc. Chim. [3], **8**, 639, 640. W. F.

564. Vedrödi, V.—*Analysis of tobacco.—Estimation of nicotin.*

The author finds fault with both Kosutány's and Kissling's method for the estimation of nicotin.

Ztschr. anal. Chem., **32**, 277–296; Jour. Chem. Soc., **64**, A, 504. J. W. F.

APPARATUS.

565. Gooch, F. A., 1892.—*Some convenient forms of laboratory apparatus.*

The author describes several convenient appliances, including a burette clip and mercury washer.

Am. Jour. Sci., 1892, 239–242. J. W. F.

566. Thörner, W.—*The use of centrifugal machines in analytical and microscopical work.*

Chem. Ztg., **16** (1892), 1101–1104; Analyst, **17**, 230–232; Exp. Sta. Record, **4**, 288. F. W. W.

567. Lehmann, M., 1894.—*A mill for preparing oil-bearing seeds, etc., for fat determinations (Extractionsmühle).*

Chem. Ztg., **18**, 412, 413; Chem. Centralbl., 1894, i, 874, 875. J. P. S.

568. Voorhees, E. B., and Voorhees, L. A.—*Some new forms of apparatus.*

A water oven for drying in hydrogen may be briefly described as a double-walled water oven of copper opening at the top by a heavy brass lid which screws down upon a rubber gasket, and with the inner wall removable from the outer for the more convenient opening and closing of the apparatus. One of the openings for the passage of hydrogen is through the lid, the other through the side near the bottom. Fodders are dried in fat extraction tubes with a plug of asbestos laid on the bottom as in Gooch crucibles. Hydrogen is supplied from a Kipp generator, provided with a simple device for securing a constant supply.

Apparatus for fat extraction consists of 12 extractors, each complete with separate lamp, bath, condenser, etc., united into one piece of apparatus. The condensers are of iron piping 1¼ inches in diameter, united by piping three-fourths inch in diameter. A single stream of water effects the cooling.

A bath for digestion of phosphates in ammonium citrate is made of copper and is provided with a frame notched for holding the flasks.

Pipettes of special volume for the simplification of calculation are made to deliver, when definite weights of substances are made up to definite volumes, an amount of the original substance equal to the conversion factor divided by an integer, which latter, by cancellation, becomes the multiplier.

For the determination of—	Weight of substance taken.	Volume of solution.	Volume of pipette.	Substance delivered by pipette.	Conversion factor.	New factor.
	Grams.	*cc.*	*cc.*	*Grams.*		
Total phosphoric acid	2	200	63.965	0.63965	63.965	100
Insoluble phosphoric acid	2	100	63.965	1.27930	63.995	50
Soluble phosphoric acid	2	300	47.974	.31983	63.965	200
Potash	10	500	48.270	.96540	19.308	20
Chlorin	10	500	49.452	.98904	24.276	25

An apparatus for moving wash solutions through the agency of compressed air is described.

Jour. anal. Chem., **7**, 121. A. L. W.

569. MULLER, J. A.—*A new gas-measuring apparatus* (*Sur un nouveau gaz-volumètre*).

For details, see original.

Bull. Soc. Chim. [3], **7**, 507–511. W. F.

570. FUCHS, F.—*A new combustion furnace.*

The furnace, which is described with the aid of drawings, combines in principle Erlenmeyer's and Babo's systems. The author claims to have accomplished this in such a manner that while the advantages of each are retained, their disadvantages are excluded.

Ber. chem. Ges., **25**, 2723–2725; Jour. Chem. Soc., **62**, 1514. J. W. F.

571. SAUER, E.—*New drying apparatus for organic analysis.*

Ber. chem. Ges., **25**, 258; Bull. Soc. Chim., [3] **8**, 670, 671. W. F.

572. REID, A. F.—*Improvements in burettes.*

A metal scale constructed to slide on the outside of an ordinary burette, or a graduated tube fixed firmly in an India-rubber ring, which slides up and down as a piston, is suggested as useful for measuring the delivery for successive small quantities of solution from the same burette.

Chem. News, **65**, 125; Jour. Chem. Soc., **62**, 1027. J. W. F.

573. DOOLITTLE, O. S.—*The torsion viscometer.*

An apparatus similar in general construction and method of operation to that earlier described by Babcock. (Report N. Y. State Agr. Exp. Station.)

Drugs, Oils, and Paints, 1893, June; Jour. Am. Chem. Soc., **15**, 1893, 173–177.

574. FÉRY, CH.—*A new refractometer* (*Un nouveau réfractomètre*).

Essentially a spectroscope, 2-tube, with a hollow prism replaced. When the prism is empty the monochromatic light passes through both tubes. On introduction of a liquid, refraction displaces the ray. By lateral movement of the prism the coincidence is restored. The amount of this displacement serves as a measure of the refractive index. A very little liquid (2 cc.) is enough for the test. Cross lines in both telescopes render great accuracy possible.

La Nature; Rev. internat. falsificat., **7**, 7–9. F.

575. VIVIEN, September, 1892.—*Vacuum drying apparatus* (*Étuve à vide*).

The apparatus described and illustrated in detail provides for the rapid drying in vacuo of various sugar products. In this drying chamber provision is also made for the collection of the water which is evolved, and for the disposal of the volatile matters given off during the operation of drying.

Bull. Assoc. Chim. France, **10**, 343.

576. SPENCER, G. L., May, 1893.—*An air-drying oven.*

For details see original. The essential features consist in double walls filled on sides and top with a nonconducting solid, on bottom with air; drying flasks, mounted in a circle inside the orifice, connected with a circular tube, in turn connected with a mercury exhaust pump. These flasks have suspended in the neck water traps, preventing any water of condensation from falling back into the flask. The air in the oven is stirred by a revolving fan connected with a small motor; the whole is heated by a circular gas lamp, with mercury regulator. By a separate series of tubes hydrogen or other gas may be passed through the stoppers into the drying flasks.

Jour. Am. Chem. Soc., **15**, 82–84. W. F.

577. SAUER, E., 1894.—*A new form of air bath.*

Jour. Am. Chem. Soc., **16**, 31–33. J. P. S.

578. STOLBA, F.—*A distillation apparatus for ammonia determinations* (*Apparat zur Bestimmung von Ammoniak durch Destillation*).

The apparatus, which is described and illustrated in detail, consists of a globular flask, a bulbed tube, and a U-shaped receiver. The tube which connects the receiver with the rest of the apparatus passes up through a sealed connection in the large bulb, reaching nearly to the top of the latter, while the bulb itself is directly connected by a tube with the distillation flask. It is claimed that with this apparatus no errors are occasioned by any portion of the contents of the flask being carried over into the receiver. No standard acid is placed in the receiver at first, the ammonia being collected in water to which the indicator has been added, and the standard acid being run in at intervals through the open arm of the U-tube, the contents of the tube being agitated at the same time. In this way it is claimed that the end of the evolution of ammonia can be readily determined. The receiver is constantly surrounded by cold water.

Cas. pro. prum. chem., **3**, 94; Chem. Ztg. Rep., **17**, 111. B. B. R.

579. MÜLLER, M.—*A safe distilling apparatus, especially for use in the Kjeldahl method*

Ztschr. Angew. Chem., 1893, 229, 230; Chem. Centralbl., 1893, **2**, 3.

A.—APPARATUS FOR SOIL WORK.

580. TACKE, B.—*Apparatus for determining the volume of soil samples* (*Ein Volumenometer für die Ermittelung des Volumens grösserer Proben, besonders von Bodenproben*).

The author describes his somewhat elaborate apparatus and gives formulæ for the calculation of results.

Ztschr. angew. Chem., 1893, 39; Ber. chem. Ges., **26**, Ref. 335, A. L. W.

581. MARVIN, C. F., January, 1892.—*Description of an improved soil thermometer.*

This instrument is a thermometer designed to be more sensitive than ordinary registering thermometers, and to avoid the errors due to their usually large and variable bore. It consists of a long, slender-bored stem, prolonged nearly to the bottom of the bulb, which here has a small spherical enlargement. The principal part of the bulb is filled with alcohol, but the small spherical enlargement and most of the tube contain mercury, over which a little alcohol column stands. Ordinary steel indexes are used; the maximum reading is given by the index pushed up by expanding mercury; the minimum by a correction of the reading indicated by the upper index pulled downward by the contracting alcohol; the indexes are set by a magnet. In very long thermometers the ungraduated part of the stem is made much smaller in bore than the graduated portion.

Agric. Science, **7**, 28–31. W. F.

582. Kedzie, R. C., August, 1892.—*A simple evaporimeter.*

The apparatus described consists of a small metallic cup, with perpendicular sides, into which a small tube enters horizontally near the surface of the soil or water contained in the cup; this tube is connected by a rubber tube, closed by a pinch-cock, with a burette. At the close of any set interval of time the cups are filled from the burette to original level or original degree of wetness, as evinced by touch or appearance, and reading of burette shows amount of water evaporated.

Agric. Science, **6**, 473, 474. W. F.

B.—Apparatus for sugar work.

583. Wiley, H. W., 1893.—*Lamp for a constant monochromatic flame.*

Consists of two cylinders of platinum gauze, mounted parallel, between which the flame burns; these revolve in opposite directions by means of clockwork, and dipping into solutions of salt in porcelain crucibles constantly bring fresh portions of evenly distributed salt to the flame.

Jour. Am. Chem. Soc., **15**, 121, 123. W. F.

584. Pellet, H., September, 1892.—*The continuous polarization tube* (*Le tube continu pour la polarisation de tous les liquides sucrés*).

Reference is made to the various modifications of the original continuous polarization tube, notably those recently proposed by Loehr, Stift, and others. The author states that the tube devised by him, and its modifications, have given general satisfaction in many laboratories, the tubes being free from trouble with air bubbles and providing for rapid and expeditious rinsing and filling. Moreover, these tubes are more simple in construction than those above referred to.

Sucr. indig., **40**, 359. B. B. R.

585. Casteels, O., March, 1893.—*Pellet's continuous polarization tube* (*Notice sur les tube continu H. Pellet*).

The Pellet continuous polarization tube is highly commended for rapidity and ease of manipulation and accuracy of the results secured. The author gives results of tests of beet pulp, diffusion juice, scums, etc., employing both the ordinary tube and that of Pellet's, the figures obtained being closely concordant throughout. A number of experiments showed that with Pellet's 400 mm. tube, 100 cc. of juice will suffice to rinse the tube and give a correct polarization, though the quantity of liquid required varies with the diameter of the tube. After polarizing a sample from 90 to 110 cc. of water are found sufficient to reduce the reading to zero.

Bull. Assoc. Chim. Belge., **6**, 263. B. B. R.

C.—Estimation of fat.

586. Graftiau, March, 1893.—*New fat extractor* (*Nouvelle extracteur pour le dosage des matières grasses*).

The apparatus, of which a full description and illustration are given, has the advantage of possessing very few connections, the extractor proper having only one, and that being a mercury joint. The flask which collects the extract is not connected directly with the extractor tube, but is inclosed in an enameled metallic cylinder, provided with a grooved collar, into which the outer tube of the extractor fits, the connection being sealed by means of mercury.

Bull. Assoc. Chim. France, **10**, 646. B. B. R.

587. Farsteiner, K., July, 1892.—*A simple condensing and extracting apparatu* (*Eine einfache Kühl- und Extractions-Vorrichtung*).

The apparatus described and illustrated is quite simple in its construction and differs from the ordinary extraction apparatus chiefly as regards the condenser. The large outer tube, inside of which is placed the tube containing the material to be extracted, is provided with a large perforated stopper through which pass the arms of a bent tube 0.5 cm. in diameter. Through this bent tube a constant stream of cold water is kept flowing, the vapor of the liquid employed in extraction being condensed by contact with the cool surface of the tube. The simplicity of the apparatus, and the small number of connections necessary, are among its chief advantages.

Chem. Ztg., **16,** 1030; Vjschr. Chem. Nahr., **1,** 360. B. B. R.

588. HOLDE.—*Extraction apparatus* (*Extractions-Apparat zum Extrahiren von Flüssigkeiten und breiigen Substanzen*).

The apparatus described is designed for the extraction of liquids or substances of a thick, pasty consistency, and is constructed somewhat upon the principle of the Soxhlet extractor. The ether vapor from the flask passes up through a side tube to a condenser, and, after condensation, drops into a funnel tube, which conveys it to the bottom of the tube containing the substance to be extracted; the ether passes up through this substance and the ethereal layer is siphoned off when it reaches a certain height. A cut illustrates the mode of operation of the apparatus.

Mitth. kgl. tech. Versuchss. Berlin, **10,** 130; Chem. Ztg. Rep., **16,** 275.

B. B. R.

589. WILLARD, J. T., and FAILYER, G. H.—*Extraction apparatus.*

This differs but little from the usual siphon extractor. The substance to be extracted is placed in a "pocket" on the side of the extractor, and a movable siphon tube withdraws the extract when the pocket becomes filled.

Jour. anal. Chem., **5,** No. 8; Chem. Centralbl., 1892, i, 4; Jour. Chem. Soc., **62,** 910. J. W. F.

590. WILEY, H. W.—*A new extraction apparatus.*

The apparatus described and figured is a simplification of Knorr's apparatus. The metal condenser consists of four double conical sections, in each section of which is a circular disk which causes the water entering at the bottom to pass against the inner metal surface. The vapor of ether comes in contact with the outer surface of the condenser. A Gooch crucible suspended from the condenser holds the substance to be extracted. Both condenser and crucible are inclosed in a large test tube ground at the mouth to fit tightly against a turned brass plate. The extract may be collected either in the bottom of the test tube or in a flask made to fit into the test tube.

Jour. anal. Chem., **7,** 65; Jour. Am. Chem. Soc., **15,** 123-126. A. L. W.

591. BÜTTNER, W.—*A simple extraction apparatus for analytical work* (*Einfacher Extraktionsapparat für analytische Arbeiten*).

The apparatus consists of two test tubes, one placed within the other, a reflux condenser and a capillary siphon. The outer test tube should be 150 to 160 mm. long and 30 to 35 mm. wide, and should be heated a little below the middle and drawn out to form a neck on which the inner tube may rest. The inner tube contains the substances to be extracted in a capsule of filter paper, and the ether is withdrawn from its bottom by means of a capillary siphon. The outer tube is connected with the condenser by means of the only cork which is used in the apparatus.

Ztschr. angew. Chem., 1893, 634, 635. W. D. B.

592. BARTELS, L.—*Extraction apparatus.*

This apparatus consists of a reflux condenser, funnel, and wide-neck flask. The funnel, containing a weighed filter and the substance to be extracted, is suspended from the condenser in such a manner that the ether drops into it from the condenser. The flask passes over the funnel and fits a cork on the lower end of the condenser tube. The amount of extract may be taken either from the gain in weight of the flask or the loss in weight of the funnel.

Ztschr. angew. Chem., 1893, 525; Vjschr. Chem. Nahr., 1893, **8,** 318. W. D. B.

593. Étaix, L.—*A new extraction apparatus.*
Bull. Soc. Chim., **11** [3], 259; Ber. chem. Ges., **27**, Ref. 425.

594. Carcano, L.—*A new modification of the Soxhlet extractor.*
Staz. sper. agr. Ital., **24**, 234, 235; Chem. Centralbl., 1893, **2**, 129.

D.—MILK AND BUTTER APPARATUS.

595. Bartlett, J. M.—*Testing butter.*

The author describes a bottle for use in testing butter by the Babcock milk test. The bottle is similar to the cream bottle, except that a bulb is blown in the middle of the neck to enable it to hold the butter fat. The range of readings is from 70 to 90 per cent of fat, using 18 grams of butter. The efficiency of the bottle has not yet been tested.

Maine Sta. Bull. 4 (2d ser.), 12, fig. 1; Exp. Sta. Record, **4**, 11. E. W. A.

596. Gerber, N.—*Acidbutyrometer for fat determinations.*
Chem. Ztg. **16**, 1839; Chem. Centralbl., 1893, 233.

597. Drouot.—*Apparatus for the examination of butter.*

The apparatus is based on the difference in the appearance of natural and artificial butters in melting. Besana and Wollny give a favorable opinion of the method, while Rava and Salvatori express themselves unfavorably to the apparatus for detection of mixtures below 20 per cent of adulteration.

Analyst, **17**, 100. F. W. W.

TANNIN.

598 Snyder, H.—*Notes on Löwenthal's method.*
Jour. Am. Chem. Soc., **15**, 560-562.

599. Procter, H. R.—*Qualitative determination of tannins.*
Jour. Am. Chem. Soc., **16**, 247.

600. Procter, H. R.—*The technical tanning materials, especially gambier, analysis of.*
Jour. Soc. Chem. Ind., xi, 329.

601. Wood, J. T.—*Fermentation in the leather industry.*
Jour. Soc. Chem. Ind., xiii, 3, 218.

602. Procter, H. R.—*Note on the estimation of tanning matter.*
Jour. Soc. Chem. Ind., xiii, 5, 494.

603. Canaigre as a substitute for barks.
U. S. Consular Reports, 1894, 44; Jour. Soc. Chem. Ind., xiii, 6, 650.

604. Trimble and Peacock.—*Canaigre tannin.*
Am. Jour. Pharm., xxiii, 4, 161.

605. Trimble and Peacock.—*The preparation of oak tannin, with special reference to the use of acetone as a solvent.*
Am. Jour. Pharm., xxiii, 9, 435.

606. Hooper, David.—*Myrica Nagi Thun.*
Am. Jour. Pharm. xxiv, 5, 209.

607. Manger, Ch. C.—*Myrica asplenifolia.*
Am. Jour. Pharm., 1894, 66, 211; Chem. Ztg., xviii, 58, 186.

608. Culley, John.—*The tannin of Pumica granatum.*
Am. Jour. Pharm. xxiv, 6, 280.

609. Thompson, Wm. B.—*Oak wood and bark.*
Am. Jour. Pharm., xxiv, 6, 293.

610. Trimble, Henry.—*Oak barks from India.*

Am. Jour. Pharm., xxiv, 6, 299.

611. Hooper, David.—*Indian tanning materials.*
Am. Jour. Pharm. xxiv, 8, 377.

612. Haenlein, F. H.—*Origin of acid fermentation in tanning liquors.*
Dingl. polyt. Jour., 1894, **291**, 186–190.

613. Sadlon, C.—*The course of absorption in the tannin layers.*
Dingl. polyt. Jour., 1893, **288**, 256.

614. von Schröder.—*Practical extraction of myrobalans.*
Dingl. polyt. Jour., 1894, 75, 213; Chem. Ztg., xviii, 52, 169.

615. Schroeder and Bartel.—*Extraction of tanning materials.*
Dingl. polyt. Jour., 1894, **291**, 259–263.

616. Fahrion, Dr. W.—*The theory of the formation of leather.*
Chem. Ztg., xvii, 23, 394.

617. Jean, F.—*Analysis of tannin extracts.*
Chem. Ztg., xvii, 27, 470.

618. Pässler, Dr. J.—*Does tannin tan?*
Chem. Ztg., xviii, 21, 363.

619. Fölsing, Dr. A.—*Progress in the field of tanning and tanning substances.*
Chem. Ztg., xviii, 96, 1770.

620. Eitner, W.—*The influence of light, heat, and fermentation on the composition of tanning substances.*
Der Gerber, **13**, 245.

621. Morpurgo, G.—*Estimation of tannin.*
Der Gerber, **18**, 288; Ztschr. anal. Chem., **32**, 617.

622. Weiss, B.—*Criticism of the use of lead carbonate for estimating tannin.*
Der Gerber, **18**, 288.

623. Bartel, A.—*Purification of hide powder.*
Deutsche Gerberztg., **35**, No. 56.

624. Proctor.—*Solubilities of tannin.*
Deutsch-Amerik. Apoth.-Ztg., **10**, 276; Ztschr. anal. Chem., **32**, 616.

625. Trimble, Henry.—*Examination of the tannin of chestnut bark.*
Proc. Frank. Inst., **4**, 23.

626. Flückiger, F. A.—*Notes on tannins.*
Pharm. Post, **27**, 233.

627. Fleury, G.—*The estimation of tannin.*
Jour. de Phys. et de Chim. (5 ser.), **25**, 499; Bull. Soc. Chim., Paris (3 ser.), **7**, 687.

628. Gawalosky, A.—*Estimation of tannic acid, gallic acid, and pectin.*
Brestewski's Handwörterbuch der Pharmacie.

629.—*Production of gambier in India.*
Pharm. J. Trans. (1894), 1228, 54.

TABLE OF ATOMIC MASSES OF THE ELEMENTS.

Revised by F. W. Clarke, chief chemist of the U. S. Geological Survey, to January 1, 1894. (Wiley's Agricultural Analysis, p. 3.)

Name.	Symbol.	Atomic weight.	Name.	Symbol.	Atomic weight.
Aluminum	Al	27	Neodymium	Nd	140.5
Antimony	Sb	120	Nickel	Ni	58.7
Arsenic	As	75	Nitrogen	N	14.03
Barium	Ba	137	Osmium	Os	190.8
Bismuth	Bi	208.9	Oxygen[3]	O	16
Boron	B	11	Palladium	Pd	106.6
Bromin	Br	79.95	Phosphorus	P	31
Cadmium	Cd	112	Platinum	Pt	195
Cesium	Cs	132.9	Potassium	K	39.11
Calcium	Ca	40	Praseodymium	Pr	143.5
Carbon	C	12	Rhodium	Rh	103
Cerium	Ce	140.2	Rubidium	Rb	85.5
Chlorin	Cl	35.45	Ruthenium	Ru	101.6
Chromium	Cr	52.1	Samarium	Sm	150
Cobalt	Co	59	Scandium	Sc	44
Columbium[1]	Cb	94	Selenium	Se	79
Copper	Cu	63.6	Silicon	Si	28.4
Erbium	Er	166.3	Silver	Ag	107.92
Fluorin	F	19	Sodium	Na	23.05
Gallium	Ga	69	Strontium	Sr	87.6
Germanium	Ge	72.3	Sulphur	S	32.06
Glucinum[2]	Gl	9	Tantalum	Ta	182.6
Gold	Au	197.3	Tellurium	Te	125
Hydrogen	H	1.007	Terbium	Tb	159.5
Indium	In	113.7	Thallium	Tl	204.18
Iodin	I	126.85	Thorium	Th	232.6
Iridium	Ir	193.1	Tin	Sn	119
Iron	Fe	56	Titanium	Ti	48
Lanthanum	La	138.2	Tungsten	W	184
Lead	Pb	206.95	Uranium	U	239.6
Lithium	Li	7.02	Vanadium	V	51.4
Magnesium	Mg	24.3	Ytterbium	Yb	173
Manganese	Mn	55	Yttrium	Yt	89.1
Mercury	Hg	200	Zinc	Zn	65.3
Molybdenum	Mo	96	Zirconium	Zr	90.6

[1] Has priority over Niobium. [2] Has priority over Beryllium. [3] Standard or basis of the system.

OFFICIAL METHODS OF ANALYSIS ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS AT ITS MEETING, AUGUST 23, 24, 25, 1894.

METHODS FOR PHOSPHORIC ACID.

PREPARATION OF SAMPLE.

The sample should be well intermixed, finely ground, and passed through a sieve having circular perforations 1 mm. in diameter. The process of grinding and sifting should take place as rapidly as possible, so that there should be no loss or gain of moisture during the operation.

DETERMINATION OF MOISTURE.

(*a*) In potash salts, sodium nitrate, and ammonium sulphate, heat from 1 to 5 grams at 130° until the weight is constant, and determine the loss in weight as moisture. (*b*) In all other fertilizers, heat 2 grams, or 5 grams if the sample be very coarse, for five hours, at 100°, in a steam bath.

METHODS OF DETERMINING PHOSPHORIC ACID.

Water-soluble phosphoric acid.—Place 2 grams in a 9 cm. filter; wash with successive small portions of water, allowing each portion to pass through before adding more, until the filtrate measures about 250 cc. If the filtrate be turbid, add a little nitric acid. Make up to any convenient definite volume; mix well; take any convenient portion and determine the phosphoric acid as under total phosphoric acid.

Citrate-insoluble phosphoric acid.—Heat 100 cc. of strictly neutral ammonium-citrate solution of 1. 0900 sp. gr. to 65° C. in a flask placed in a bath of warm water, keeping the flask loosely stoppered to prevent evaporation. When the citrate solution in the flask has reached 65° C., drop it into the filter containing the washed residue from the water-soluble phosphoric-acid determination, stopper tightly with a smooth rubber stopper, and shake violently until the filter paper is reduced to a pulp. Place the flask back into the bath and maintain the water in the bath at such a temperature that the contents of the flask will stand at exactly 65° C. Shake the flask every five minutes. At the expiration of exactly thirty minutes from the time the filter and residue were introduced, remove the flask from the bath and immediately filter as rapidly as possible. Wash thoroughly with water at 65° C. Transfer the filter and its contents to a crucible, ignite until all organic matter is destroyed, add from 10 to 15 cc. of strong hydrochloric acid, and digest until all phosphate is dissolved, or return the filter with contents to the digestion flask, add 30 to 35 cc. strong nitric acid and 5 to 10 cc. strong hydrochloric acid, and boil until all phosphate is dissolved. Dilute the solution to 200 cc. If desired, the filter and its contents can be treated according to methods 1, 2, or 3 under total phosphoric acid. Mix well; filter through a dry filter; take a definite portion of the filtrate and determine phosphoric acid as under total phosphoric acid.

In case a determination of citrate-insoluble phosphoric acid is required in nonacidulated goods it is to be made by treating 2 grams of the phosphatic material, without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, etc.) the residue insoluble in ammonium citrate is to be treated by one of the processes described below under "total phosphoric acid" 1, 2, or 3.

Total phosphoric acid.—In case of ignition the ignited material is to be dissolved in hydrochloric acid. Weigh 2 grams and treat by one of the following methods: (1) Evaporate with 5 cc. of magnesium nitrate, ignite, and dissolve in hydrochloric acid. (2) Boil with 20 to 30 cc. of strong sulphuric acid, adding 2 to 4 grams of sodium or potassium nitrate at the beginning of the digestion and a small quantity after the solution has become nearly colorless, or adding the nitrate in small portions from time to time. A Kjeldahl flask graduated to 250 cc. is recommended. After the solution is colorless, add 150 cc. of water and boil for a few minutes, cool, and make up to volume. (3) Digest with strong sulphuric acid and such other reagents as are used in either the plain or modified Kjeldahl or Gunning methods for estimating nitrogen. Do not add any potassium permanganate, but after the solution has become colorless add about 100 cc. of water and boil for a few minutes, cool, and make up to a convenient volume; 2.5 grams of substance and a digestion flask graduated to 250 cc. are recommended. [This method will be found convenient when both the nitrogen and the total phosphoric acid are to be determined in a fertilizer. In this case, after diluting the volume and mixing, a portion, for the estimation of nitrogen, may be removed with a pipette and the remainder then filtered through a dry filter and a portion taken for the determination of the total phosphoric acid.] (4) Solution in 30 cc. of concentrated nitric acid with a small quantity of hydrochloric acid. (5) Add 30 cc. of concentrated hydrochloric acid, heat, and add cautiously, in small quantities at a time, about 0.5 grams of finely-pulverized potassium chlorate. (6) Solution in 15 to 30 cc. of strong hydrochloric acid and 3 to 10 cc. of nitric acid. This method is recommended for fertilizers containing much iron or aluminum phosphate. Boil until all phosphates are dissolved and all organic matter is destroyed; cool and dilute to 200 or 250 cc.; mix and pass through a dry filter; take an aliquot part of the filtrate corresponding to 0.25 gram, 0.50 gram, or 1 gram, neutralize with ammonia, and clear with a few drops of nitric acid. (In case hydrochloric or sulphuric acid has been used as a solvent, add about 15 grams of dry ammonium nitrate or its equivalent.)

To the hot solutions, for every decigram of P_2O_5 that is present, add 50 cc. of molybdic solution. Digest at about 65° C. for an hour, filter, and wash with water or ammonium-nitrate solution. Test the filtrate by renewed digestion and addition of more molybdic solution. Dissolve the precipitate on the filter with ammonia and hot water and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (about 1 drop per second), stirring vigorously. After fifteen minutes add 30 cc. of ammonia solution of density 0.95. Let stand for some time; two hours is usually enough. Filter, wash with dilute ammonia, ignite at white heat for ten minutes, and weigh.

Citrate-soluble phosphoric acid.—The sum of the water soluble and citrate insoluble subtracted from the total gives the citrate soluble.

PREPARATION OF REAGENTS.

(1) *Ammonium-citrate solution.*—(*a*) Mix 370 grams of commercial citric acid with 1,500 cc. of water; nearly neutralize with commercial ammonia; cool; add ammonia until exactly neutral (testing with saturated alcoholic solution of corallin) and bring to volume of 2 liters. Test the specific gravity, which should be 1.0900 at 20° C., before using.

(*b*) *Alternate method.*—To 370 grams of commercial citric acid add commercial ammonia, sp. gr. 0.96, until nearly neutral; reduce the specific gravity to nearly 1.09 and make exactly neutral, testing as follows: Prepare a solution of fused calcium chlorid, 200 grams to the liter, and add 4 volumes of strong alcohol. Make the mixture exactly neutral, using a small amount of freshly-prepared corallin solution as preliminary indicator, and test finally by withdrawing a portion, diluting with an equal volume of water, and testing with cochineal solution; 50 cc. of this solution will precipitate the citric acid from 10 cc. of the citrate solution. To 10 cc. of the nearly neutral citrate solution add 50 cc. of the alcoholic calcium-chlorid solution, stir well, filter at once through a folded filter, dilute with an equal volume of water, and test the reaction with neutral solution of cochineal. If acid or alkaline, add ammonia or citric acid, as the case may be, mix, and test again, as before. Repeat this process until a neutral reaction is obtained. Bring to the proper specific gravity of 1.0900 at 20° C.

(2) *Molybdic solution.*—Dissolve 100 grams of molybdic acid in 400 grams or 417 cc. of ammonia, sp. gr. 0.96, and pour the solution thus obtained into 1,500 grams or 1,250 cc. of nitric acid, sp. gr. 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phosphomolybdate. Decant the solution from any sediment and preserve in glass-stoppered vessels.

(3) *Ammonium-nitrate solution.*—Dissolve 200 grams of commercial ammonium nitrate in water and bring to a volume of 2 liters.

(4) *Magnesia mixture.*—Dissolve 22 grams of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding an excess of the latter. Add a little calcined magnesia in excess, and boil a few minutes to precipitate iron, alumina, and phosphoric acid; filter; add 280 grams of ammonia chlorid, 700 cc. of ammonia of sp. gr. 0.96, and water enough to make a volume of 2 liters. Instead of the solution of 22 grams of calcined magnesia, 110 grams of crystallized magnesium chlorid ($MgCl_2$ $6H_2O$) may be used.

(5) *Dilute ammonia for washing.*—This solution shall be prepared so as to contain 2½ per cent NH_3.

(6) *Nitrate of magnesia.*—Dissolve 320 grams of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess; boil; filter from the excess of magnesia, ferric oxid, etc., and bring to volume of 2 liters.

METHODS FOR THE DETERMINATION OF NITROGEN.

THE ABSOLUTE OR CUPRIC OXID METHOD.

[Applicable to all nitrogen determinations.]

The apparatus and reagents needed are as follows:

APPARATUS.

Combustion tube of best hard Bohemian glass, about 66 cm. long and 12.7 mm. internal diameter.

Azotometer of at least 100 cc. capacity, accurately calibrated.

Sprengel mercury air pump.

Small paper scoop, easily made from stiff writing paper.

REAGENTS.

Coarse cupric oxid or wire form.—To be ignited and cooled before using.

Fine cupric oxid.—Prepared by pounding ordinary cupric oxid in a mortar.

Metallic copper.—Granulated copper, or fine copper gauze, reduced and cooled in a current of hydrogen.

Sodium bicarbonate.—Free from organic matter.

Caustic potash solution.—Make a supersaturated solution of caustic potash in hot water. When absorption of carbonic acid ceases to be prompt the solution must be discarded.

MANIPULATION.

Filling the tube.—Of ordinary commercial fertilizers take 1 to 2 grams for analysis. In the case of highly nitrogenized substances the amount to be taken must be regulated by the amount of nitrogen estimated to be present. Fill the tube as follows: (1) About 5 cm. of coarse cupric oxid. (2) Place on the small paper scoop enough of the fine cupric oxid to fill, after having been mixed with the substance to be analyzed, about 10 cm. of the tube; pour on this the substance, rinsing the watch glass with a little of the fine oxid, and mix thoroughly with a spatula; pour into the tube, rinsing the scoop with a little fine oxid. (3) About 30 cm. of coarse cupric oxid. (4) About 7 cm. of metallic copper. (5) About 6 cm. of coarse cupric oxid (anterior layer). (6) A small plug of asbestos. (7) 0.8 to 1 gram of sodium bicarbonate. (8) A large, loose plug of asbestos; place the tube in the furnace, leaving about 2.5 cm. of it projecting; connect with the pump by a rubber stopper smeared with glycerol, taking care to make the connection perfectly tight.

OPERATION.

Exhaust the air from the tube by means of the pump. When a vacuum has been obtained allow the flow of mercury to continue; light the gas under that part of the tube containing the metallic copper, the anterior layer of cupric oxid (see 5th above), and the sodium bicarbonate. As soon as the vacuum is destroyed and the apparatus filled with carbonic acid, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer just below the surface of the mercury seal, so that the escaping bubbles will pass into the air and not into the tube, thus avoiding the useless saturation of the caustic-potash solution.

When the flow of carbonic acid has very nearly or completely ceased, pass the delivery tube down into the receiving arm, so that the bubbles will escape into the azotometer. Light the gas under the 30 cm. layer of oxid, heat gently for a few moments to drive out any moisture that may be present, and bring to red heat. Heat gradually the mixture of substance and oxid, lighting one jet at a time. Avoid a too rapid evolution of bubbles, which should be allowed to escape at the rate of about one per second or a little faster.

When the jets under the mixture have all been turned on, light the gas under the layer of oxid at the end of the tube. When the evolution of gas has ceased, turn out all the lights except those under the metallic copper and anterior layer of oxid, and allow to cool for a few moments. Exhaust with the pump and remove the azotometer before the flow of mercury is stopped. Break the connection of the tube with the pump, stop the flow of mercury, and extinguish the lights. Allow the azotometer to stand for at least an hour, or cool with a stream of water until a permanent volume and a temperature have been reached.

Adjust accurately the level of the KOH solution in bulb to that in the azotometer; note the volume of gas, temperature, and height of barometer; make calculation as usual, or read results from tables.

THE KJELDAHL METHOD.

[Not applicable in the presence of nitrates.]

REAGENTS.

(1) *Acid.*—(*a*) Standard hydrochloric acid, the absolute strength of which has been determined by precipitating with silver nitrate and weighing the silver chlorid as follows:

To any convenient quantity of the acid to be standardized, add solution of silver nitrate in slight excess, and 2 cc. pure nitric acid, specific gravity, 1.2. Heat to boiling point, and keep at this temperature for some minutes without allowing violent ebullition, and with constant stirring, until the precipitate assumes the granular form. Allow to cool somewhat, and then pass the fluid through the asbestos. Wash the precipitate by decantation, with 200 cc. of very hot water, to which has been added 8 cc. nitric acid and 2 cc. dilute solution of silver nitrate containing 1 gram of the salt in 100 cc. of water. The washing by decantation is performed by adding the hot mixture in small quantities at a time, and beating up the precipitate well with a thin glass rod after each addition. The pump is kept in action all the time, but to keep out dust during the washing the cover is only removed from the crucible when the fluid is to be added.

Put the capsule and precipitate aside, return the washings once through the asbestos so as to obtain them quite clear, remove them from the filter, and set aside to recover excess of silver. Rinse the receiver and complete the washing of the precipitate with about 200 cc. of cold water. Half of this is used to wash by decantation and the remainder to transfer the precipitate to the crucible with the aid of a trimmed feather. Finish washing in the crucible, the lumps of silver chlorid being broken down with the glass rod. Remove the second filtrate from the receiver and pass about 20 cc. of 98 per cent alcohol through the precipitate. Dry at 140° to 150°. Exposure for half an hour is found more than sufficient, at this temperature, to dry the precipitate thoroughly.

Or (*b*) standard sulphuric acid the absolute strength of which has been determined by precipitation with barium chlorid and weighing the resulting barium sulphate.

For ordinary work half normal acid is recommended, *i. e.*, acid containing 18.2285 grams of hydrochloric acid or 24.5185 grams sulphuric acid to the liter; for work in determining very small amounts of nitrogen, one-tenth normal acid is recommended. In titrating mineral acids against ammonia solutions, use cochineal as indicator.

(2) *Standard alkali*, the strength of which, relative to the acid, has been accurately determined. One-tenth normal ammonia solution, *i. e.*, containing 1.7051 grams of ammonia to the liter, is recommended for accurate work.

(3) *Sulphuric acid*, specific gravity 1.84, free from nitrates and also from ammonium sulphate, which is sometimes added in the process of manufacture to destroy oxids of nitrogen.

(4) *Metallic mercury* or mercuric oxid, prepared in the wet way. That prepared from mercuric nitrate can not be safely used.

(5) *Potassium permanganate* finely pulverized.

(6) *Granulated zinc*, pumice stone, or 0.5 gram of zinc dust are to be added to the contents of the flasks in distillation, when found necessary, in order to prevent bumping.

(7) *Potassium sulphid.*—A solution of 40 grams of commercial potassium sulphid in 1 liter of water.

(8) *Soda.*—A saturated solution of sodium hydrate free from nitrates.

(9) *Indicator.*—Solution of cochineal prepared as follows: Tincture of cochineal is prepared by digesting and frequently agitating 3 grams of pulverized cochineal in a mixture of 50 cc. of strong alcohol with 200 cc. of distilled water, at ordinary temperatures, for a day or two. The solution is decanted or filtered through Swedish paper.

APPARATUS.

(1) *Kjeldahl digestion flasks* of hard, moderately thick, well-annealed glass. These flasks are about 22 cm. long, with a round, pear-shaped bottom, having a maximum diameter of 6 cm., and tapering out gradually in a long neck, which is 2 cm. in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 cc.

(2) *Distillation flasks* of ordinary shape, of 550 cc. capacity, or preferably flasks of the same capacity, of pear-shaped bottom, of well-annealed glass, for both digestion and distillation, fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation.

The bulbs are about 3 cm. in diameter, the tubes being of the same diameter as the condenser and cut off obliquely at the lower end. This is adjusted to the tube of the condenser by a rubber tube.

MANIPULATION.

(1) *The digestion.*—0.7 to 3.5 grams of the substance to be analyzed, according to its proportion of nitrogen, is brought into a digestion flask with approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury and 20 cc. of sulphuric acid. The flask is placed in an inclined position, and heated below the boiling point of the acid for from five or fifteen minutes or until frothing has ceased. If the mixture froths badly, a small piece of paraffin may be added to prevent it. The heat is then raised until the acid boils briskly. No further attention is required till the contents of the flask have become a clear liquid, which is colorless or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and while still hot, potassium permanganate is dropped in carefully and in small quantities at a time till, after shaking, the liquid remains of a green or purple color.

(2) *The distillation.*—After cooling, the contents of the flask are transferred to the distilling flask with about 200 cc. of water, with a few pieces of granulated zinc, pumice stone, or 0.5 gram of zinc dust when found necessary to keep the contents of the flask from bumping, and 25 cc. of potassium-sulphid solution are added, shaking the flask to mix its contents. Next add 50 cc. of the soda solution, or sufficient to make the reaction strongly alkaline, pouring it down the side of the flask so that it does not mix at once with acid solution. Connect the flask with the condenser, mix the contents by shaking, and distil until all ammonia has passed over into the standard acid. The first 150 cc. of the distillate will generally contain all the ammonia. This operation usually requires from forty minutes to one hour and a half. The distillate is then titrated with standard alkali.

The use of mercuric oxid in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in case of substances most difficult to oxidize, and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. The potassium sulphid removes all the mercury from the solution, and so prevents the formation of mercur-ammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present, which might otherwise escape notice.

GUNNING METHOD.

MANIPULATION.

In a digestion flask holding from 250 to 500 cc., place from 0.7 to 3.5 grams of the substance to be analyzed, according to its proportion of nitrogen. Then add 10 grams of powdered potassium sulphate and from 15 to 25 cc. (ordinarily about 20 cc.) of concentrated sulphuric acid. Conduct the digestion as in the Kjeldahl process, starting with a temperature below boiling point and increasing the heat gradually until frothing ceases. Digest until colorless or nearly so. (Do not add either potassium permanganate or potassium sulphid.) Dilute, neutralize, and distil as in the Kjeldahl method. In neutralizing, it is convenient to add a few drops of phenolphthalein indicator, by which one can tell when the acid is completely neutralized, remembering that the pink color, which indicates an alkaline reaction, is destroyed by a considerable excess of strong fixed alkali. The distillation and titration are conducted as in the Kjeldahl method.

KJELDAHL METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.

[Applicable to all fertilizers containing nitrates.]

Besides the reagents and apparatus given under the Kjeldahl method there will be needed—

(1) *Zinc dust.* This should be an impalpable powder; granulated zinc or zinc filings will not answer.

(2) *Sodium thiosulphate.*

(3) *Commercial salicylic acid.*

DETERMINATION.

Bring from 0.7 to 3.5 grams of the substance to be analyzed into a Kjeldahl digesting flask, add to this 30 cc. of sulphuric acid containing 1 gram of salicylic acid and shake until thoroughly mixed with the acid, then add 5 grams of crystallized sodium thiosulphate; or add to the substance 30 cc. of sulphuric acid containing 2 grams of salicylic acid, then add gradually 2 grams of zinc dust, shaking the contents of the flask at the same time. Finally place the flask on the stand for holding the digestion flasks, where it is heated over a low flame until all danger from frothing has passed. The heat is then raised until the acid boils briskly and the boiling continued until white fumes no longer pour out of the flask. This requires about five or ten minutes. Add now approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury, and continue the boiling until the liquid in the flask is colorless or nearly so. (In case the contents of the flask are likely to become solid before this point is reached add 10 cc. more of sulphuric acid.) Complete the oxidation with a little potassium permanganate in the usual way and proceed with the distillation as described in the Kjeldahl method. The reagents should be tested by blank experiments.

GUNNING METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.

MANIPULATION.

In a digestion flask holding from 250 to 500 cc. place from 0.7 to 3.5 grams of the substance to be analyzed, according to the amount of nitrogen present. Add 30 to 35 cc. of salicylic-acid mixture, namely, 30 cc. sulphuric acid to 1 gram of salicylic acid, shake until thoroughly mixed and allow to stand five to ten minutes, with frequent shaking; then add 5 grams thiosulphate of sodium and 10 grams of potassium sulphate. Heat very gently until frothing ceases, then heat strongly until nearly colorless. Dilute, neutralize, and distil the same as in the Gunning method.

ALTERNATE METHODS.

Ruffle Method.

REAGENTS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) A mixture of equal parts by weight of fine slaked lime and finely powdered sodium thiosulphate dried at 100°.

(3) A mixture of equal parts by weight of finely powdered granulated sugar and flowers of sulphur.

(4) Granulated soda-lime, as described under the soda-lime method.

APPARATUS.

(5) Combustion tubes of hard Bohemian glass, 30 cm. long and 1.3 cm. in diameter.

(6) Bulbed U-tubes or Will's bulbs, as described under the soda-lime method.

MANIPULATION.

(1) Clean the U-tube and introduce 10 cc. of standard acid.

(2) Fit the cork and glass connecting tube. Fill the tube as follows: (1) A loosely fitting plug of asbestos, previously ignited, and then 2.5 to 3.5 cm. of the thiosulphate mixture. (2) The weighed portion of the substance to be analyzed is intimately mixed with from 5 to 10 grams of the sugar and sulphur mixture. (3) Pour on a piece of glazed paper or in a porcelain mortar a sufficient quantity of thiosulphate mixture to fill about 25 cm. of tube; then add the substance to be analyzed, as previously prepared, mix carefully, and pour into the tube; shake down the contents of the tube; rinse off the paper or mortar with a small quantity of the thiosulphate mixture and pour into the tube; then fill up with soda-lime to within 5 cm. of the end of the tube. (4) Place another plug of ignited asbestos at the end of the tube and close with a cork. (5) Hold the tube in a horizontal position and tap on the table until there is a gas channel all along the top of the tube. Make connections with the U-tube containing the acid; aspirate and see that the apparatus is tight.

The combustion.—Place the prepared combustion tube in the furnace, letting the open end project a little, so as not to burn the cork. Commence by heating the soda-lime portion until it is brought to a full red heat. Then turn on slowly jet after jet toward the outer end of the tube, so that the bubbles come off two or three a second. When the whole tube is red hot and the evolution of the gas has ceased and the liquid in the U-tube begins to recede toward the furnace, attach the aspirator to the other limb of the U-tube, break off the end of the tube, and draw a current of air through for a few minutes. Detach the U-tube and wash the contents into a beaker or porcelain dish; add a few drops of the cochineal solution, and titrate.

SODA-LIME METHOD.

[Not applicable in presence of nitrates.]

REAGENTS AND APPARATUS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) Granulated soda lime, fine enough to pass a 2.5 mm. sieve and thoroughly dry.

(3) Fine soda lime, fine enough to pass a 1.25 mm. sieve, also thoroughly dry.

Excellent soda lime may be easily and cheaply prepared by slaking 2½ parts of quicklime with a strong solution of 1 part of commercial caustic soda (such soda as is used in the Kjeldahl process), care being taken that there is enough water in the solution to slake the lime. The mixture is then dried and heated in an iron pot to incipient fusion, and when cold, ground and sifted as above.

Instead of soda lime Johnson's mixture of carbonate of soda and lime or slaked lime may be used.

Slaked lime may be granulated by mixing it with a little water to form a thick mass, which is dried in the water oven until hard and brittle. It is then ground and sifted as above. Slaked lime is much easier to work with than soda lime and gives excellent results, though it is probable that more of it should be used in proportion to the substance to be analyzed than is the case with soda lime.

(4) Asbestos which has been ignited and kept in a glass-stoppered bottle.

(5) Combustion tubes about 40 cm. long and of 12 mm. internal diameter, drawn out to a point and closed at one end.

(6) Large-bulbed U-tubes with glass stopcock, or Will's tubes with four bulbs.

MANIPULATION.

The substance to be analyzed should be powdered fine enough to pass through a sieve of 1 mm. mesh; 0.7 to 1.4 grams, according to the amount of nitrogen present is taken for the determination. Into the closed end of the combustion tube put a small loose plug of asbestos, and upon it about 4 cm. of fine soda lime. In a porcelain dish or mortar mix the substance to be analyzed, thoroughly but quickly, with enough fine soda lime to fill about 16 cm. of the tube, or about 40 times as much soda lime as substance, and put the mixture into the combustion tube as quickly as possible by means of a wide necked funnel, rinsing out the dish and funnel with a little more fine soda lime, which is to be put in on top of the mixture. Fill the rest of the tube to about 5 cm. of the end with granulated soda lime, making it as compact as possible by tapping the tube gently while held in a nearly upright position during the filling. The layer of granulated soda lime should not be less than 12 cm. long. Lastly, put in a plug of asbestos about 2 cm. long, pressed rather tightly, and wipe out the end of the tube to free it from adhering soda lime.

Connect the tube by means of a well-fitting rubber stopper or cork with the U-tube or Will's bulbs, containing 10 cc. of standard acid, and adjust it in the combustion furnace so that the end projects about 4 cm. from the furnace, supporting the U-tube or Will's bulb suitably. Heat the portion of the tube containing the granulated soda lime to a moderate redness, and when this is attained extend the heat gradually through the portion containing the substance, so as to keep up a moderate and regular flow of gases through the bulbs, maintaining the heat of the first part until the whole tube is heated uniformly to the same degree. Keep up the heat until gases have ceased bubbling through the acid in the bulbs, and the mixture of substance and soda lime has become white, or nearly so, which shows that the combustion is finished. The combustion should occupy about three-quarters of an hour or not more than one hour. Remove the heat, and when the tube has cooled below redness break off the closed tip and aspirate air slowly through the apparatus for two or three minutes to bring all the ammonia into the acid. Disconnect, wash the acid into a beaker or flask, and titrate with the standard alkali.

During the combustion the end of the tube projecting from the furnace must be kept heated sufficiently to prevent the condensation of moisture, yet not enough to char the stopper. The heat may be regulated by a shield of tin slipped over the projecting end of the combustion tube.

It is found very advantageous to attach a Bunsen valve to the exit tube, allowing the evolved gases to pass out freely, but preventing a violent "sucking back" in case of a sudden condensation of steam in the bulbs.

METHODS OF DETERMINING POTASH.

METHOD OF LINDO-GLADDING.

(1) *In superphosphates.*—Boil 10 grams with 300 cc. of water thirty minutes. To the hot solution add ammonia in slight excess, and then a sufficient quantity of ammonium oxalate to precipitate all the lime present; cool and make up to 500 cc., mix thoroughly, and filter through a dry filter; take 50 cc., corresponding to 1 gram, evaporate nearly to dryness, add 1 cc. of dilute sulphuric acid (1 to 1), evaporate to dryness and ignite to whiteness. As all the potash is in form of sulphate, no loss need be apprehended by volatilization of potash, and a full red heat must be maintained until the residue is perfectly white. This residue is dissolved in hot water, plus a few drops of hydrochloric acid, and a slight excess of platinum solution is now added. This solution is then evaporated to a thick paste in a small dish, and 80 per cent alcohol added. In evaporating, special precaution should be taken to prevent absorption of ammonia. The precipitate is washed thoroughly with alcohol by decantation and on the filter, as usual. The washing should be continued even after the filtrate is colorless. Ten cubic centimeters of the ammonium chlorid solution, prepared as hereafter directed, are now run through the filter, or the washing may be performed in the dish. The 10 cc. will contain the bulk of the impurities, and are thrown away. Fresh portions of 10cc. of the ammonium chlorid are now run through the filter several times (five or six). The filter is then washed thoroughly with pure alcohol, dried, and weighed as usual. Care should be taken that the precipitate is perfectly soluble in water. The platinum solution used contains 1 gram of metallic platinum in every 10 cc. To prepare the washing solution of ammonium chlorid,

place in a bottle 500 cc. of water, 100 grams of ammonium chlorid, shake till dissolved. Now, pulverize 5 or 10 grams of potassium platinic chlorid put in the bottle, and shake at intervals for six or eight hours; let settle over night, then filter off the liquid into a second bottle. The first bottle is then ready for preparation of a fresh supply when needed.

(2) *Muriates of potash.*—In the analysis of these salts an aliquot portion containing 0.5 grams is evaporated with 10 cc. of the platinum solution and a few drop-hydrochloric acid and washed as before.

(3) *Sulphate of potash, kainit, etc.*—In the analysis of kainit, dissolve 10 grams of the pulverized salt in 300 cc. of boiling water, add ammonia to slight excess, then a sufficient quantity of ammonium oxalate to throw down all lime present; cool and make up to 500 cc., mix thoroughly, and filter on a dry filter; take 25 cc., corresponding to 0.5 grams, and proceed to remove the ammonia, as in the analysis of superphosphates; dissolve the residue in hot water plus a few drops of hydrochloric acid and add 15 cc. of platinum solution. In the analysis of high-grade sulphate and of double-manure salt (potassium sulphate, magnesium sulphate, containing about 27 per cent of potassium oxid), make up the solution as above, but omit the precipitation, evaporation, etc.; take an aliquot part equal to 0.5 gram and add 15 cc. of platinum solution. In all cases special care must be taken in the washing with alcohol to remove all the double platinum-sodium chlorid if present. The washing should be continued some time after the filtrate is colorless. Twenty-five cubic centimeters of the ammonium chlorid solution are employed instead of 10 cc., and the 25 cc. poured through at least six times to remove all sulphates and chlorids. Wash finally with alcohol; dry and weigh as usual.

ALTERNATE METHOD FOR POTASH.

Take 10 grams of the prepared sample, boil for 30 minutes with 300 cc. of water, and, after cooling and without filtering, make up to 1,000 cc. and filter through a dry filter. If the sample has 10 per cent of potassium oxid, take 50 cc. of the filtrate; if less than 10 per cent of potassium oxid (ordinary potash fertilizers), take 100 cc. of the filtrate. In each case make the volume up to 150 cc., heat to 100°, and add, drop by drop with constant stirring, slight excess of barium chlorid, without filtering; in the same manner add barium hydrate in slight excess. Filter while hot and wash until the precipitate is free of chlorids. Add to the filtrate 1 cc. of strong ammonium hydrate, and then a saturated solution of ammonium carbonate until the excess of barium is precipitated. Heat; add now, in fine powder, 0.5 grams pure oxalic acid or 0.75 grams of ammonium oxalate. Filter; wash free of chlorids, evaporate the filtrate to dryness in a platinum dish, and ignite carefully over the free flame below red heat until all volatile matter is driven off.

The residue is now digested with hot water, filtered through a small filter, and washed with successive small portions of water until the filtrate amounts to 30 cc. or more. To this filtrate, after adding 2 drops of strong hydrochloric acid, are added, in a porcelain dish, 5 to 10 cc. of a solution of 10 grams of platinic chlorid in 100 cc. of water. The mixture is now evaporated on a water bath to a thick sirup, as above, treated with 80 per cent alcohol, washed by decantation, collected in a Gooch crucible or other form of filter, washed with strong alcohol, afterwards with 5 cc. of ether, dried for 30 minutes at 100°, and weighed.

It is desirable, if there be an appearance of foreign matter in the double salt, that it should be washed, according to the previous method, with 10 cc. of the half-concentrated solution of ammonium chlorid, which has been saturated by shaking with potassium platinic chlorid.

METHOD OF SOLUTION FOR ORGANIC COMPOUNDS.

In case the potash is contained in organic compounds like tobacco stems, cottonseed hulls, etc., weigh out 10 grams, saturate with strong sulphuric acid, and ignite in a muffle to destroy organic matter. Add a little strong hydrochloric acid to moisten the mass and warm slightly so as to loosen it in the dish. Proceed then as in the Lindo-Gladding or alternate method.

FACTORS.

The use of the factors 0.3056 for converting potassium platinic chlorid to potassium chlorid and 0.19308 for converting to potassium oxid is continued.

METHODS OF SUGAR ANALYSIS.*

DETERMINATION OF WATER.

In sugar or masse cuite.—Dry from 2 to 5 grams in a flat dish (nickel or platinum) at the temperature of boiling water for ten hours; cool in a desiccator and weigh; return to the oven and dry for an hour. If on weighing there is only a slight change of weight, the process may be considered finished; otherwise the drying must be continued until the loss of water in one hour is not greater than 0.20 per cent.

In honey or molasses.—Proceed as above, using only 1 to 2 grams of substance.

The density of juices, sirups, etc., is most conveniently determined by means of Baumé's or Brix's hydrometer or areometer, preferably with the latter, as the graduations of the scale give close approximations to the percentages of total solids. The Brix spindle should be graduated to tenths. It is therefore desirable, for accuracy, that the range of degrees recorded by each individual spindle be as limited as possible, this end being best secured by the employment of sets, consisting of not less than three spindles. The solutions should be as nearly as possible of the same temperature as the air at the time of reading, and if the variation from the standard temperature of the graduation of the spindle amounts to more than 1°, compensation therefor must be made by reference to tables of corrections for temperature.

Care should be observed in taking the density of a juice to allow it to stand in the cylinder until all air bubbles have escaped, before reading the spindles. When it is desired to accurately determine the specific gravity of the sample, a specific-gravity bottle or pyknometer is employed.

The per cent of total solids can be approximately ascertained directly from the reading of the Brix hydrometer, or, knowing the reading of the Baumé spindle or the specific gravity of the sample, by reference to the table on page 214, Bulletin 31.

DETERMINATION OF ASH.

(*a*) Heat 5 or 10 grams of the material (sugar, molasses, honey) in a platinum dish† of 50 to 100 cc. capacity at 100° until the water is expelled, and then slowly over a flame until intumescence ceases. The dish is then placed in a muffle and heated at low redness until a white ash is obtained.

For soluble ash digest the ash with water, filter through a Gooch crucible, wash with hot water, dry the residue at 100°, and weigh. The difference of weights equals soluble ash.

(*b*) Use 50 milligrams of zinc oxid to 25 grams of molasses or 50 grams of sugar.

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[*] By the term "sugar analysis" it is understood that the analyses of all the common saccharine substances, viz, juices, sugar, molasses, sirup, starch sugar, and honey are embraced. In the following directions an attempt will be made to indicate the general principles which should guide the practice of sugar analysis, together with sufficient detail to enable these analyses to be carried out with accuracy and uniformity.

The principal components to be determined in the substances mentioned above are:

1. Water.
2. Density and total solids.
3. Ash.
4. Nitrogenous bodies.
5. Reducing sugar.
6. Sucrose.
7. Lactose.
8. Starch sugar.
9. Raffinose.

[†] If the substance contains tin or any other metal capable of uniting with platinum, a dish made of some other material must be used.

Incorporate thoroughly by adding dilute alcohol and mixing; dry and ignite as above. Deduct the weight of zinc used from the weight of ash.

(*c*) Carbonize the mass at a low heat, dissolve the soluble salts with hot water, burn the residual mass as above, add the solution of soluble salts, and evaporate to dryness at 100°; ignite gently, cool in a desiccator, and weigh.

(*d*) Saturate the sample with H_2SO_4, dry, ignite gently, then burn in a muffle at low redness. Deduct one-tenth of the weight of the ash, then calculate the per cent.

(*e*) Dissolve 10 grams of sugar in hot water and filter to remove sand, etc.; evaporate the filtrate and washings to dryness; carefully carbonize and extract with hot water until there is no longer any reaction for chlorin. Dry and ignite the residue; weigh as insoluble ash. Add the soluble portion, treat with HCl in slight excess, and evaporate to dryness. Heat until all free HCl is driven off; take up with water and a little HCl, filter, and wash. The residue is silica. Add NH_4OH to the filtrate, boil and filter, wash the residue, which is iron oxid and alumina. To the filtrate add $(NH_4)_2C_2O_4$; evaporate to dryness; ignite and moisten with $(NH_4)_2CO_3$ and reignite; dissolve in water, filter, and wash. The residue on the filter represents calcium and magnesium carbonate. Evaporate the filtrate to a small bulk, add $(NH_4)_2CO_3$, and evaporate to dryness; drive off the excess of ammonia cautiously and weigh. This gives the alkalis as carbonates, and this weight, added to the insoluble ash, formerly determined, gives the total carbonate ash.

(*f*) Thoroughly mix 5 grams of the material with a somewhat larger weight of pure quartz sand in a platinum dish; ignite in a muffle at a moderate red heat.

ALTERNATE METHOD FOR ASH.

To avoid the correction of one-tenth, as proposed by Scheibler, and one-fifth as proposed by Girard and Violette, when sugars are burned with H_2SO_4, Boyer suggests incineration with benzoic acid, as giving the real quantity of mineral matter without correction.

The benzoic acid is dissolved in alcohol of 90 per cent, 25 grams of the acid to 100 cc. of alcohol; 5 grams of the sugar are weighed in a capsule and moistened with 1 cc. of water. The capsule is heated slowly in order to caramelize the sugar without carbonizing it; 2 cc. of the benzoic-acid solution are next added, and the capsule warmed until all the alcohol is evaporated; the temperature is then raised until the sugar is converted into carbon. The decomposing benzoic acid produces abundant vapors, which render the mass extremely porous, especially if a circular motion is imparted to the capsule. The slow heating is continued until all the benzoic acid is volatilized.

The carbon obtained is voluminous and of a brilliant black color. The incineration is accomplished in a muffle at a low red heat. The capsule should be weighed quickly when taken from the desiccator, in order to avoid the absorption of water by the alkaline carbonates. Ammonium benzoate may be employed instead of benzoic acid, and the analyst should previously assure himself that neither the acid nor the ammonia salt leaves a residue on incineration. In addition to giving the mineral matters directly, this method permits the determination of their composition also, a matter of no small importance.

DETERMINATION OF NITROGEN.

Any of the methods adopted by the association for the estimation of nitrogen may be used.

REDUCING SUGARS.

Reagents (Soxhlet's modification of Fehling's solution).

(1) 34.639 grams of $CuSO_4, 5H_2O$, dissolved and diluted to 500 cc. with water.

(2) 173 grams of Rochelle salt, 50 grams of NaOH, dissolved and diluted to 500 cc. with water. Mix equal volumes of these solutions immediately before use. This

mixture is the "mixed copper reagent" to be used for all the methods given below except Allihn's method for dextrose, for which a special reagent must be used.

(*a*) *Approximate volumetric method for rapid work.*—Place 10 cc. of the mixed copper reagent in a large test tube and add 10 cc. of distilled water. Heat to boiling, and gradually add small portions of the solution of the material to be tested, boiling to complete the reaction after each addition, until the copper has been completely precipitated. Two minutes' boiling is required for complete precipitation when the full amount of sugar solution has been added in one portion. When the end reaction is nearly reached and the amount of sugar solution to be added can no longer be judged by the color of the solution, a small portion of the liquid is removed by means of Knorr's modification of Wiley's filtering tube (any other rapid means of filtration may be used that removes but a small portion of the hot liquid), is transferred to a small porcelain crucible or test plate, acidified with dilute acetic acid, and tested for copper with a dilute solution of potassium ferrocyanid. The sugar solution should be of such a strength as will give a burette reading of 15 to 20 cc., and the number of successive additions should be as small as possible.

Since the factor for calculation varies with the minute details of manipulation, every operator must determine a factor for himself, using a known solution of a pure sample of the sugar that he desires to determine and keeping the conditions the same as those used for the determinations. For the standardization of a solution for the determination of invert sugar in sugar-house products, dissolve 2.5 grams of pure sucrose in 100 cc. of water, add 10 cc. of hydrochloric acid (specific gravity 1.188), and invert according to the method given below for double polarization. Neutralize the acid with sodium carbonate and dilute to 1 liter. The 2.5 grams of sucrose become 2.6316 grams of invert sugar. The weight of invert sugar equivalent to 10 cc. of the copper reagent is calculated as follows:

$$\frac{2.6316 \times \text{by number of cc. of the standard sugar solution used}}{1000} = X,$$

the weight of invert sugar required to completely precipitate the copper in 10 cc. of the reagent under the conditions used for the test titration. For the calculation of the result of the titration of an unknown solution:

Let X = the factor obtained as above;

V = the number of cc. of unknown sugar solution required to precipitate the copper from 10 cc. of copper solution;

W = the weight of the material under examination in 1 cc. of the solution.

Then $\frac{100X}{VW}$ = per cent of invert sugar in the sample.

The calculation can be much simplified by so standardizing the copper reagent that 50 milligrams of invert sugar will be required to reduce the copper from 10 cc. of the copper reagent. The various tables given in works on sugar analysis then become applicable. These tables are arranged for a "glucose normal solution" containing 5 grams of the material to be examined in 100 cc. When the weight per 100 cc. is more or less than 5 grams the number found in the table is increased or diminished accordingly.

(*b*) *Soxhlet's exact volumetric method.*—A preliminary titration is made to determine the approximate percentage of reducing sugar in the material under examination. A solution is then prepared which contains approximately 1 per cent of reducing sugar. Place in a beaker 100 cc. of the mixed copper reagent and approximately the amount of the sugar solution for its complete reduction. Boil for two minutes. Filter through a plaited filter and test a portion of the filtrate for copper by use of acetic acid and potassium ferrocyanid. Repeat the test, varying the volume of sugar solution, until two successive amounts of sugar solution are found which differ by 0.1 cc., one giving complete reduction and the other leaving a small amount of copper in solution. The mean of these two readings is taken as the volume of the solution required for the complete precipitation of 100 cc. of the copper reagent.

Under these conditions 100 cc. of the mixed copper reagent require 0.475 gram of anhydrous dextrose, or 0.494 gram of invert sugar, for complete reduction. The percentage is calculated by the following formula:

W = the weight of the sample in 1 cc. of the sugar solution;

V = the volume of the sugar solution required for the complete reduction of 100 cc. of the copper reagent.

Then $\frac{100 \times 0.475}{VW}$ = per cent of dextrose,

or $\frac{100 \times 0.494}{VW}$ = per cent of invert sugar.

(*c*) *Gravimetric method for materials containing 1 per cent or less of invert sugar.*—The solution of the material to be examined is so prepared as to contain 20 grams in 100 cc., and it must be freed from suspended impurities by filtration through paper or by use of lead acetate, removing the excess of lead by means of sodium carbonate. In a beaker of 250 cc. capacity place 50 cc. of the mixed copper reagent and 50 cc. of the sugar solution. Heat this mixture at such a rate that approximately four minutes are required to bring it to the boiling point, and boil for exactly two minutes. Add 100 cc. of cold, recently boiled, distilled water. Filter immediately through a weighed filtering tube of hard glass under pressure. The asbestos film in the filtering tube is supported by a perforated disk or cone of platinum and should be washed free from loose fibers before weighing and moistened previous to the filtration. The tube is provided with a detachable funnel during the filtration, so that none of the precipitate accumulates near the top, where it could be removed by the cork used during the reduction of the cuprous oxid. The precipitate is all transferred to the filter and thoroughly washed with hot water, following the water by alcohol and ether successively. After being dried the tube is connected with an apparatus for supplying a continuous current of dry hydrogen, gently heated until the cuprous oxid is completely reduced to the metallic state, cooled in the current of hydrogen, and weighed. The increase in weight is the weight of copper reduced by 10 grams of the sample. The corresponding percentage of invert sugar is found by use of the following table:

Herzfeld's table for the determination of invert sugar in materials containing 1 per cent or less of invert sugar.[1]

Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.
Milligrams.	*Per cent.*	*Milligrams.*	*Per cent.*	*Milligrams.*	*Per cent.*
50	0.05	120	0.40	190	0.79
55	.07	125	.43	195	.82
60	.09	130	.45	200	.85
65	.11	135	.48	205	.88
70	.14	140	.51	210	.90
75	.16	145	.53	215	.93
80	.19	150	.56	220	.96
85	.21	155	.59	225	.99
90	.24	160	.62	230	1.02
95	.27	165	.65	235	1.05
100	.30	170	.68	240	1.07
105	.32	175	.71	245	1.10
110	.35	180	.74		
115	.38	185	.76		

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[1] Zeitschrift des Vereins für die Rübenzucker-Industrie des Deutschen Reichs, 1885, **35**, 967.

The weight of copper may be obtained as follows: The filtration after reduction is made in a Gooch crucible, and the beaker and precipitate thoroughly washed with hot water without any effort to transfer the precipitate to the filter. Wash the asbestos film and the adhering cuprous oxid into the beaker by means of a wash bottle containing hot dilute nitric acid. After the copper is all in solution refilter through a Gooch crucible with a thin film of asbestos and wash thoroughly with hot water. Add 10 cc. of dilute sulphuric acid (containing 200 cc. of sulphuric acid—sp. gr. 1.84—per liter) and evaporate the filtrate on the steam bath until the copper salt has largely crystallized out. Heat carefully on a hot plate or over a piece of asbestos board until the evolution of the white fumes shows that the excess of nitric acid is removed. Add 8 to 10 drops of nitric acid (sp. gr. 1.42) and rinse into a platinum dish of 100 to 125 cc. capacity. Precipitate the copper by electrolysis. Wash with water thoroughly before breaking the current, remove the dish from the circuit, wash with alcohol and ether successively, dry at a temperature that can be borne by the hand, and weigh. The evaporation of the liquid for the removal of the excess of nitric acid may be avoided by the following process: Transfer the asbestos film from the crucible to the beaker by means of a glass rod and rinse the crucible with about 30 cc. of a boiling mixture of dilute nitric and sulphuric acids, containing 65 cc. of sulphuric acid (sp. gr. 1.84) and 50 cc. of nitric acid (sp. gr. 1.42) per liter; or the asbestos film with the adhering cuprous oxid may be treated by the method of Formanek given below, by transferring to the beaker containing the rest of the suboxid and washing the crucible with 2 to 4 cc. of nitric acid (strong) to remove all adhering precipitate, receiving the solutions in the beaker. The crucible is then washed with 5 to 10 cc. water, which is also received in the beaker. After all the cuprous oxid in the beaker is dissolved the crucible is washed with an additional quantity of water. Refilter through a Gooch crucible and wash thoroughly with hot water, limiting the volume of the filtrate to 75 to 80 cc. Transfer to a platinum dish and electrolyze as described above. Wash before interrupting the current, using water acidulated with sulphuric acid at first.

(*d*) *Gravimetric method for materials containing more than 1 per cent of invert sugar.*—Prepare a solution of the material to be examined in such a manner that it contains 20 grams in 100 cc. after clarification and the removal of the excess of lead. Prepare a series of solutions in large test tubes by adding 1, 2, 3, 4, 5, etc., cc. of this solution to each successively. Add 5 cc. of the mixed copper reagent to each, heat to boiling, boil two minutes, and filter. Note the volume of sugar solution which gives the filtrate lightest in tint, but still distinctly blue. Place twenty times this volume of the sugar solution in a 100 cc. flask, dilute to the mark, and mix well. Use 50 cc. of the solution for the determination, which is conducted as described under (*c*) until the weight of copper is obtained. For the calculation of the result use the following formulas and table of factors of Meissl and Hiller:

Let Cu = the weight of copper obtained;
P = the polarization of the sample;
W = the weight of the sample in the 50 cc. of the solution used for determination;
F = the factor obtained from the table for conversion of copper to invert sugar;

$\frac{Cu}{2}$ = approximate absolute weight of invert sugar = Z;

$Z \times \frac{100}{W}$ = approximate per cent of invert sugar = y;

$\frac{100\,P}{P+y}$ = R, relative number for sucrose;

100—R = I, relative number for invert sugar;

$\frac{Cu\,F}{W}$ = per cent of invert sugar.

Z facilitates reading the vertical columns; and the ratio of R to I, the horizontal columns of the table, for the purpose of finding the factor (F) for calculation of copper to invert sugar.

Example: The polarization of a sugar is 86.4, and 3.256 grams of it (W) are equivalent to 0.290 gram of copper. Then:

$$\frac{Cu}{2} = \frac{.290}{2} = 0.145 = Z$$

$$Z \times \frac{100}{W} = 0.145 \times \frac{100}{3.256} = 4.45 = Y$$

$$\frac{100\ P}{P + y} = \frac{8640}{86.4 + 4.45} = 95\ 1 = R$$

$$100 - R = 100 - 95.1 = I = 4.9$$

$$R : I = 95.1 : 4.9$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to Z, 145, and the horizontal column headed 95:5 is nearest to the ratio of R to I, 95.1:4.9. Where these columns meet we find the factor 51.2, which enters into the final calculation:

$$\frac{Cu\ F}{W} = \frac{0.290 \times 51.2}{3.256} = 4.56 \text{ per cent of invert sugar.}$$

Meissl and Hiller's factors for the determination of more than 1 per cent of invert sugar.[1]

Ratio of sucrose to invert sugar = R : I.	Approximate absolute weight of invert sugar = Z.						
	200 milligrams.	175 milligrams.	150 milligrams.	125 milligrams.	100 milligrams.	75 milligrams.	50 milligrams.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
0 : 100	56. 4	55. 4	54. 5	53. 8	53. 2	53	53
10 : 90	56. 3	55. 3	54. 4	53. 8	53. 2	52. 9	52. 9
20 : 80	56. 2	55. 2	54. 3	53. 7	53. 2	52. 7	52. 7
30 : 70	56. 1	55. 1	54. 2	53. 7	53. 2	52. 6	52. 6
40 : 60	55. 9	55	54. 1	53. 6	53. 1	52. 5	52. 4
50 : 50	55. 7	54. 9	54	53. 5	53. 1	52. 3	52. 2
60 : 40	55. 6	54. 7	53. 8	53. 2	52. 8	52. 1	51. 9
70 : 30	55. 5	54. 5	53. 5	52. 9	52. 5	51. 9	51. 6
80 : 20	55. 4	54. 3	53. 3	52. 7	52. 2	51. 7	51. 3
90 : 10	54. 6	53. 6	53. 1	52. 6	52. 1	51. 6	51. 2
91 : 9	54. 1	53. 6	52. 6	52. 1	51. 6	51. 2	50. 7
92 : 8	53. 6	53. 1	52. 1	51. 6	51. 2	50. 7	50. 3
93 : 7	53. 6	53. 1	52. 1	51. 2	50. 7	50. 3	49. 8
94 : 6	53. 1	52. 6	51. 6	50. 7	50. 3	49. 8	48. 9
95 : 5	52. 6	52. 1	51. 2	50. 3	49. 4	48. 9	48. 5
96 : 4	52. 1	51. 2	50. 7	49. 8	48. 9	47. 7	46. 9
97 : 3	50. 7	50. 3	49. 8	48. 9	47. 7	46. 2	45. 1
98 : 2	49. 9	48. 9	48. 5	47. 3	45. 8	43. 3	40
99 : 1	47. 7	47. 3	46. 5	45. 1	43. 3	41. 2	38. 1

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[1] Zeitschrift des Vereins für die Rübenzucker-Industrie des Deutschen Reichs, 1889, **39**, 735.

(*e*) *Allihn's gravimetric method for the determination of dextrose.*

Reagents:

I. 34.639 grams of $CuSO_4$, $5H_2O$, dissolved in water and diluted to 500 cc.

II. 173 grams of Rochelle salts, 125 grams of KOH, } dissolved in water and diluted to 500 cc.

Place 30 cc. of the copper solution (I), 30 cc. of the alkaline tartrate solution (II), and 60 cc. of water in a beaker and heat to boiling. Add 25 cc. of the solution of the material to be examined, which must be so prepared as not to contain more than 1 per cent of dextrose, and boil for two minutes. Filter immediately without diluting and obtain the weight of copper by one of the methods given under (*c*). The corresponding weight of dextrose is found by the following table:

Allihn's table for the determination of dextrose.[1]

Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.
10	6.1	58	29.8	106	54.0	154	78.6	202	103.7
11	6.6	59	30.3	107	54.5	155	79.1	203	104.2
12	7.1	60	30.8	108	55.0	156	79.6	204	104.7
13	7.6	61	31.3	109	55.5	157	80.1	205	105.3
14	8.1	62	31.8	110	56.0	158	80.7	206	105.8
15	8.6	63	32.3	111	56.5	159	81.2	207	106.3
16	9.0	64	32.8	112	57.0	160	81.7	208	106.8
17	9.5	65	33.3	113	57.5	161	82.2	209	107.4
18	10.0	66	33.8	114	58.0	162	82.7	210	107.9
19	10.5	67	34.3	115	58.6	163	83.3	211	108.4
20	11.0	68	34.8	116	59.1	164	83.8	212	109.0
21	11.5	69	35.3	117	59.6	165	84.3	213	109.5
22	12.0	70	35.8	118	60.1	166	84.8	214	110.0
23	12.5	71	36.3	119	60.6	167	85.3	215	110.6
24	13.0	72	36.8	120	61.1	168	85.9	216	111.1
25	13.5	73	37.3	121	61.6	169	86.4	217	111.6
26	14,0	74	37.8	122	62.1	170	86.9	218	112.1
27	14.5	75	38.3	123	62.6	171	87.4	219	112.7
28	15.0	76	38.8	124	63.1	172	87.9	220	113.2
29	15.5	77	39.3	125	63.7	173	88.5	221	113.7
30	16.0	78	39.8	126	64.2	174	89.0	222	114.3
31	16.5	79	40.3	127	64.7	175	89.5	223	114.8
32	17.0	80	40.8	128	65.2	176	90.0	224	115.3
33	17.5	81	41.3	129	65.7	177	90.5	225	115.9
34	18.0	82	41.8	130	66.2	178	91.1	226	116.4
35	18.5	83	42.3	131	66.7	179	91.6	227	116.9
36	18.9	84	42.8	132	67.2	180	92.1	228	117.4
37	19.4	85	43.4	133	67.7	181	92.6	229	118.0
38	19.9	86	43.9	134	68.2	182	93.1	230	118.5
39	20.4	87	44.4	135	68.8	183	93.7	231	119.0
40	20.9	88	44.9	136	69.3	184	94.2	232	119.6
41	21.4	89	45.4	137	69.8	185	94.7	233	120.1
42	21.9	90	45.9	138	70.3	186	95.2	234	120.7
43	22.4	91	46.4	139	70.8	187	95.7	235	121.2
44	22.9	92	46.9	140	71.3	188	96.3	236	121.7
45	23.4	93	47.4	141	71.8	189	96.8	237	122.3
46	23.9	94	47.9	142	72.3	190	97.3	238	122.8
47	24.4	95	48.4	143	72.9	191	97.8	239	123.4
48	24.9	96	48.9	144	73.4	192	98.4	240	123.9
49	25.4	97	49.4	145	73.9	193	98.9	241	124.4
50	25.9	98	49.9	146	74.4	194	99.4	242	125.0
51	26.4	99	50.4	147	74.9	195	100.0	243	125.5
52	26.9	100	50.9	148	75.5	196	100.5	244	126.0
53	27.4	101	51.4	149	76.0	197	101.0	245	126.6
54	27.9	102	51.9	150	76.5	198	101.5	246	127.1
55	28.4	103	52.4	151	77.0	199	102.0	247	127.6
56	28.8	104	52.9	152	77.5	200	102.6	248	128.1
57	29.3	105	53.5	153	78.1	201	103.1	249	128.7

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[1] J. prakt. Chem., 1880, **22**, 46.

Allihn's table for the determination of dextrose.[1]—Continued.

Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.
250	129.2	293	152.7	336	176.5	379	200.8	422	225.7
251	129.7	294	153.2	337	177.0	380	201.4	423	226.3
252	130.3	295	153.8	338	177.6	381	202.0	424	226.9
253	130.8	296	154.3	339	178.1	382	202.5	425	227.5
254	131.4	297	154.9	340	178.7	383	203.1	426	228.0
255	131.9	298	155.4	341	179.3	384	203.7	427	228.6
256	132.4	299	156.0	342	179.8	385	204.3	428	229.2
257	133.0	300	156.5	343	180.4	386	204.8	429	229.8
258	133.5	301	157.1	344	180.9	387	205.4	430	230.4
259	134.1	302	157.6	345	181.5	388	206.0	431	231.0
260	134.6	303	158.2	346	182.1	389	206.5	432	231.6
261	135.1	304	158.7	347	182.6	390	207.1	433	232.2
262	135.7	305	159.3	348	183.2	391	207.7	434	232.8
263	136.2	306	159.8	349	183.7	392	208.3	435	233.4
264	136.8	307	160.4	350	184.3	393	208.8	436	233.9
265	137.3	308	160.9	351	184.9	394	209.4	437	234.5
266	137.8	309	161.5	352	185.4	395	210.0	438	235.1
267	138.4	310	162.0	353	186.0	396	210.6	439	235.7
268	138.9	311	162.6	354	186.6	397	211.2	440	236.3
269	139.5	312	163.1	355	187.2	398	211.7	441	236.9
270	140.0	313	163.7	356	167.7	399	212.3	442	237.5
271	140.6	314	164.2	357	188.3	400	212.9	443	238.1
272	141.1	315	164.8	358	188.9	401	213.5	444	238.7
273	141.7	316	165.3	359	189.4	402	214.1	445	239.3
274	142.2	317	165.9	360	190.0	403	214.6	446	239.8
275	142.8	318	166.4	361	190.6	404	215.2	447	240.4
276	143.3	319	167.0	362	191.1	405	215.8	448	241.0
277	143.9	320	167.5	363	191.7	406	216.4	449	241.6
278	144.4	321	168.1	364	192.3	407	217.0	450	242.2
279	145.0	322	168.6	365	192.9	408	217.5	451	242.8
280	145.5	323	169.2	366	193.4	409	218.1	452	243.4
281	146.1	324	169.7	367	194.0	410	218.7	453	244.0
282	146.6	325	170.3	368	194.6	411	219.3	454	244.6
283	147.2	326	170.9	369	195.1	412	219.9	455	245.2
284	147.7	327	171.4	370	195.7	413	220.4	456	245.7
285	148.3	328	172.0	371	196.3	414	221.0	457	246.3
286	148.8	329	172.5	372	196.8	415	221.6	458	246.9
287	149.4	330	173.1	373	197.4	416	222.2	459	247.5
288	149.9	331	173.7	374	198.0	417	222.8	460	248.1
289	150.5	332	174.2	375	198.6	418	223.3	461	248.7
290	151.0	333	174.8	376	199.1	419	223.9	462	249.3
291	151.6	334	175.3	377	199.7	420	224.5	463	249.9
292	152.1	335	175.9	378	200.3	421	225.1		

[1] J. prakt. Chem., 1880, **22**, 46.

(*f*) *Modified method of Formanek for electrolytic estimation of reduced copper.*—To the solution obtained by the treatment of the precipitated suboxid with nitric acid and hot water add a sufficient quantity of water to reduce the nitric acid content of the solution below 5 per cent, and electrolyze.

Instead of dissolving the suboxid with nitric acid on the filter, the asbestos filter and contents are transferred from the funnel to the beaker used in the precipitation.

and the funnel is washed out with a 4 per cent nitric-acid solution. This acid solution is added to the contents of the beaker until a bulk of about 200 cc. is attained.

Two platinum cylinders are then immersed in the liquid and connections are made with a battery, the larger cylinder being the cathode.

ESTIMATION OF SUCROSE.

(*a*) *In sugars, masse cuites, etc.*—Take the normal weight in tared dish for the instrument employed; wash into a 100 cc. flask; add water until volume is 80 to 85 cc. When the crystals are all dissolved add sufficient lead acetate to throw down all precipitable matter. With molasses and masse cuites add sufficient acetic acid to convert the sub into the neutral acetate. Make up to mark, using a little ether spray to dissolve bubbles; filter, throwing away the first 10 to 15 cc.; place in observation tube and polarize. If too dark to read, filter through finely-powdered dry bone black, rejecting the first 30 to 40 cc.

For adjusting the polariscope, graduating flasks, etc., the method of the U. S. Bureau of Internal Revenue, adopted last year, was continued. (See Bull. 31, pp. 228–232.)

(*b*) *In juices, etc.*—Transfer, by means of a pipette, to the tared sugar dish the normal, or multiple normal weight of the juice or sirup to be analyzed.

In the case of juices and thin sirups, the contents of the dish are at once washed into the 100 cc. flask.

The solution in the flask is made up to 80 or 90 cc., and the least amount of basic lead-acetate solution sufficient to decolorize the contents is added. The solution is then made up to the 100 cc. mark and thoroughly shaken before filtering. In case much foaming and bubbling takes place, rendering gauging difficult, the addition of a few drops of ether is found advantageous. The solution is then poured upon a dry filter and the filtrate is collected in a dry beaker, the first portion to pass through being rejected. If it is found impossible to obtain a perfectly clear filtrate it may be necessary to decolorize with dry powdered bone black, either mixed with the liquid itself or placed upon the filter.

In case of dark-colored products, the addition of alumina cream immediately after the lead subacetate is quite advantageous, the quantity of the former employed being in excess of that of the latter. For many sugars the use of alumina cream alone will produce a satisfactory clarification. Where double polarization is employed, acetic acid should be added to the filtrate used for the direct reading in sufficient proportion to break up the compound of lead and levulose which has been formed.

Preparation of lead subacetate solution.—Boil an aqueous solution of lead acetate with an excess of lead oxid (PbO) for half an hour, and make filtered solution of a concentration of not less than 1.25 sp. gr. Solid subacetate of lead may be substituted for the normal salt and oxid in the preparation of the solution.

Alumina cream.—Prepare a cold saturated solution of alum in water and divide into two unequal portions. Add a slight excess of ammonium hydrate to the larger portion, and then add by degrees the remaining alum solution until a faintly acid reaction is secured.

Optical method by inversion.—For raw sugar, molasses, etc.

(*a*) *Method of Clerget.*—Make up the solution as above, and place 50 cc. of the filtrate in a flask marked at 50 and 55 cc. Fill to the upper mark with pure fuming HCl and mix well; place in water and heat until the thermometer, with the bulb as near the center of the sugar solution in the flask as possible, marks 68°, consuming about ten minutes in the heating; remove, cool quickly to room temperature, and polarize, noting the temperature. If the sample contained originally any invert sugar the second polarization should be made at approximately the same temperature as the first. The percentage of sucrose is then calculated by the following formula:

$S =$ percentage of sucrose
$a =$ first polarization.
$b =$ second polarization (usually to the left).
$a \pm b =$ sum of the polarizations.
$t =$ temperature of observation.

Then

$$S = \frac{a \pm b}{144 - \frac{t}{2}}$$

a and b are to be added when of different signs and the difference taken when of the same sign.

(*b*) *The official German method* may be used as follows: 26.048 grams are dissolved in a sugar flask and the solution made up to 100 cc.; 50 cc. of this solution are transferred by means of a pipette to a 50 or 55 cc. flask, cleared, and polarized, the reading being corrected for the extra 5 cc. The liquid adhering to the pipette is washed into the 100 cc. flask containing the remaining 50 cc. (13.024 grams), 5 cc. of concentrated hydrochloric acid (38 per cent, specific gravity 1.18 at 15°) added and the flask heated for fifteen minutes at 67° to 70° in a water bath. The temperature should not exceed this limit. The flask is then cooled rapidly and the solution made up to 100 cc. If the liquid is colored, it is shaken with ½ to 1 gram of bone black and filtered through a double filter. It is then polarized in a 200-mm. tube which is provided with a thermometer. As the rotatory power of invert sugar is much influenced by the temperature, this factor must be regarded. The reading is best taken between 18° and 22° and the temperature accurately determined. The reading must also be doubled on account of the dilution of the liquid.

To calculate per cent of sucrose (R) the two readings are added together, the sum (S) multiplied by 100 and divided by $142.4 - \frac{1}{2}t$ where t is the temperature at which the invert reading was taken. If the temperature is exactly 20°, the result can be made more accurate by substituting 142.66 for 142.4, thus:

$$R = \frac{100\,S}{142.66 - \frac{20}{2}} = \frac{100\,S}{132.66} = 0.7538\,S.$$

If very much invert sugar is present, both the direct and indirect readings must be taken at the same temperature.

(*c*) *Method of Lindet.*—Place 50 cc. of the filtrate as above in a flask marked at 100.5 cc.; add 5 grams of powdered zinc (zinc dust), place in boiling water, and when at the boiling point add, little by little, 5 cc. of strongest HCl. After the acid has all been added, cool and make up to mark. Polarize in a 400-mm. tube, or multiply the reading in a 200-mm. tube by 2. Calculate the percentage of sucrose as before.

(*d*) *Gravimetric method.*—Determine first any reducing sugar in the sample; then invert sucrose, neutralize free acid, and redetermine the reducing sugar. Deduct the percentage of reducing sugar obtained at first, and the remainder will be reducing sugar derived from sucrose; multiply this number by 0.95 to obtain the percentage of sucrose in the sample.

ELECTIVE METHODS.

The elective methods remain the same as in Bulletin 28, pp. 217–228.

METHODS OF ANALYSIS OF FOODS AND FEEDING STUFFS.

PREPARATION OF SAMPLE.

The substance is to be ground and passed through a sieve with circular holes 1 mm. in diameter.

DETERMINATION OF MOISTURE.

Dry from 2 to 3 grams of the substance for five hours, at the temperature of boiling water, by passing a current of dry hydrogen through the sample. If the substance be held in a glass vessel, the latter should not be in contact with the boiling water.

DETERMINATION OF ASH.

Char 2 to 3 grams of the substance and burn to whiteness at the lowest possible red heat. If a white ash can not be obtained in this manner, exhaust the charred mass with water; collect the insoluble residue on a filter, burn, add this ash to the residue from the evaporation of the above aqueous extract, and heat the whole to a low redness till the ash is white.

DETERMINATION OF ETHER EXTRACT.

Extract 2 to 3 grams of the substance dried as for the determination of the moisture, with anhydrous and alcohol-free ether, sixteen hours. Dry the extract, by exposure to the full heat of boiling water, to constant weight.

ALTERNATE METHOD FOR ETHER EXTRACT.

In determining hygroscopic water, as above, continue the drying until the loss of weight in thirty minutes is reduced to 1 milligram or less; extract the dried substance for sixteen hours as directed, dry again and give loss of weight as ether extract.

Anhydrous ether.—To prepare the anhydrous alcohol-free ether required for estimation of fat, take any of the commercial brands of ether, wash with two or three successive portions of distilled water, add sticks of solid caustic soda or potash until most of the water has been abstracted from the ether. Carefully cleaned metallic sodium, cut into small pieces, is now added until there is no further evolution of hydrogen gas. The ether thus dehydrated must be kept over metallic sodium, and should be only lightly stoppered in order to allow any accumulating hydrogen gas to escape; and it may be drawn off with a pipette as required

CRUDE PROTEIN.

Determine nitrogen as directed for nitrogen in fertilizers and multiply the result by 6.25 for the crude protein.

DETERMINATION OF ALBUMINOID NITROGEN—STUTZER'S METHOD.

To 0.7 to 0.8 gram of the substance add 100 cc. of water in a beaker, heat to boiling, or in the case of substances rich in starch heat on the water bath ten minutes, add a quantity of cupric hydrate mixture containing 0.5 to 0.6 gram of the hydrate; stir thoroughly, filter when cold, wash with cold water, and put the filter and its contents into the concentrated sulphuric acid for the determination of nitrogen. The filter papers used must be practically free of nitrogen. Add sufficient potassium-sulphid solution to completely precipitate all copper and mercury. If the substance examined consist of seed of any kind, or residues of seeds, such as oil cake or anything else rich in alkaline phosphates, add a few cubic centimeters of a concentrated solution of alum just before adding the cupric hydrate, and mix well by stirring. This serves to decompose the alkaline phosphates. If this is not done cupric phosphate and free alkali may be formed, and the protein-copper may be partially dissolved in the alkaline liquid.

Cupric hydrate.—Prepare cupric hydrate as follows: Dissolve 100 grams of pure culpric sulphate in 5 liters of water, and add 25 cc. of glycerol; add dilute solution of sodium hydrate until the liquid is alkaline; filter; rub the precipitate up with water containing 5 cc. of glycerol per liter, and then wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10 per cent of glycerol, thus preparing a uniform gelatinous mass that can be measured out with a pipette. Determine the quantity of cupric hydrate per cubic centimeter of this mixture.

DETERMINATION OF CRUDE FIBER.

Extract 2 grams of the substance with ordinary ether, at least nearly completely, or take the residue from the determination of the ether extract. To this residue, in a 500 cc. flask, add 200 cc. of boiling 1.25 per cent sulphuric acid; connect the flask with a return-flow condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Boil at once, and continue the boiling for thirty minutes. A blast of air conducted into the flask may serve to reduce the frothing of the liquid. Filter; wash thoroughly with boiling water till the washings are no longer acid; rinse the substance back into the same flask with 200 cc. of a boiling 1.25 per cent solution of sodium hydrate at least almost free from sodium carbonate; boil at once, and continue the boiling for thirty minutes in the same manner as directed above for the treatment with acid. Filter in a Gooch crucible and wash with boiling water till the washings are neutral; dry at 110°; weigh; incinerate completely, and give the loss of weight for crude fiber.

The filter used for the first filtration may be linen, one of the forms of glass-wool or asbestos filters recommended in the last report, or any other form that secures clear and reasonably rapid filtration. The solutions of sulphuric acid and sodium hydrate are to be made up of the specified strength accurately by titration, and not merely from specific gravity.

METHODS OF ANALYSIS OF DAIRY PRODUCTS.

BUTTER.

MICROSCOPIC EXAMINATION.

Place a small portion of the fresh samples, taken from the inside of the mass, on a slide, add a drop of pure sweet oil, cover with gentle pressure, and examine with a one-half to one-eighth inch objective for crystals of lard, etc. Examine the same specimen with polarized light and a selenite plate without the use of oil. Pure fresh butter will neither show crystals nor a particolored field with selenite. Other fats melted and cooled and mixed with butter will usually present crystals and variegated colors with the selenite plate.

For further microscopic study dissolve 4 or 5 cc. of the fat in 15 cc. of ether in a test tube. Close the tube with a loose plug of cotton wool and allow to stand twelve to twenty-four hours at room temperature (20° to 25°). When crystals form at the bottom of the tube they are removed with a pipette, glass rod, or tube, placed on a slide, covered, and examined. The crystals formed by later deposits may be examined in a similar way.

SAMPLING.

If large quantities of butter are to be sampled a butter trier or sampler may be used. The portions thus drawn, about 500 grams, are to be perfectly melted in a closed vessel at as low a heat as possible, and when melted the whole is to be shaken violently for some minutes till the mass is homogeneous.* A portion is then poured into the vessel from which it is to be weighed for analysis, and should nearly or quite fill it. This sample should be kept in a cold place till analyzed.

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* The mass must be sufficiently solidified to prevent the separation of the water and fat after it is placed in the bottle.

DETERMINATION OF WATER.

One and five-tenths to 2.5 grams are dried to constant weight at the temperature of boiling water in a dish with flat bottom, having a surface of at least 20 sq. cm.

The use of clean dry sand or asbestos with the butter is admissible, and is necessary if a dish with round bottom is employed.

DETERMINATION OF FAT.

The dry butter from the water determination is dissolved in the dish with absolute ether, or with 76° benzine. The contents of the dish are then transferred to a weighed Gooch crucible with the aid of a wash bottle filled with the solvent, and are washed till free from fat. The crucible and contents are heated at the temperature of boiling water till the weight is constant. The weight of fat is calculated from the data obtained.

ALTERNATE METHOD FOR FAT.

Water may be determined by drying the butter on asbestos or sand, and the fat extracted by anhydrous alcohol-free ether. The extract, after evaporation of the ether, is heated to constant weight at the temperature of boiling water and weighed.

DETERMINATION OF CASEIN AND ASH.

The crucible containing the residue from the fat determination, consisting of casein and ash, is covered and heated, gently at first, gradually raising the temperature to just below redness. The cover may then be removed and the heat continued till the contents of the crucible are white. The loss in weight of the crucible and contents represents casein, and the residue in the crucible mineral matter. In this mineral matter, dissolved in water slightly acidulated with nitric acid, chlorin may be determined gravimetrically with silver nitrate, or volumetrically, using potassium chromate as an indicator.

DETERMINATION OF SALT.

The amount of the butter or butter substitute to be taken is from 5 to 10 grams; weigh in a counterpoised beaker glass. The butter is placed, in portions of about 1 gram at a time, in the beaker, these portions being taken from different parts of the sample. Hot water is now added (about 20 cc.) to the beaker containing the butter, and after it has melted the liquid is poured into the bulb of a separating funnel. The stopper is now inserted and the contents shaken for a few moments. After standing until the fat has all collected on top of the water, the stopcock is opened and the water is allowed to run into an Erlenmeyer flask, being careful to let none of the fat globules pass. Hot water is again added to the beaker, and the foregoing process is repeated from ten to fifteen times, using each time 10 to 20 cc. of water. The resulting washings contain all but a mere trace of the NaCl originally present in the butter. The sodium chlorid is now determined in the filtrate by a standard solution of $AgNO_3$, using a few drops of a saturated solution of potassium chromate as an indicator.

SPECIFIC GRAVITY.

Standardization of flasks.—Use a small specific-gravity flask of from 25 to 30 cc. capacity. The flask is to be thoroughly washed with hot water, alcohol, and ether, and then dried. After cooling in a desiccator, the weight of the flask and stopper is accurately determined.

The flask is filled with freshly boiled and still hot distilled water, and placed in a bath of pure distilled water. The water of the bath is kept in brisk ebullition for thirty minutes, any evaporation from the flask being replaced by the addition of boiling distilled water. The stopper* is then inserted, the flask removed, wiped dry, and after it is nearly cooled to room temperature placed in the balance, and weighed when balance temperature is reached.

Weight of fat at the temperature of boiling water.—The flask is emptied of its water, rinsed with alcohol and ether, and dried again for a few minutes at the temperature of boiling water. It is then filled with the dry, hot, fresh-filtered fat, which should be entirely free from air bubbles, and replaced in the water bath, kept for thirty minutes at the temperature of boiling water, removed, and treated as above. The weight of fat having been determined, the specific gravity is obtained by dividing it by the weight of water previously found.

Example.

	Grams.
Weight of flask No. 22, dry	10.0197
Weight of flask No. 22, plus water	37.3412
Weight of water	27.3215
Weight of flask No. 22, plus fat	34.6111
Weight of fat	24.5914

Specific gravity $=24.5914 \div 27.3215 = .90008$.

The weight of the flask dry and empty, and the weight of water at boiling temperature contained therein, may be used constantly if great care be taken in handling and cleaning the apparatus.

Example.

	Grams.
Weight of flask No. 10, dry and empty	10.0028
Weight of flask after three weeks' use	10.0030

Alternate method of standardizing flasks.—Formula for calculating the volume V, in cubic centimeters, of a glass vessel from the weight P of water of temperature t contained therein, and the volume V′ at any other temperature t' (from Landolt and Börnstein's physical-chemical tables, p. 39, Table 17):

$$V = P\frac{p}{d}$$

$$V' = P\frac{p}{d}\left[1 + \gamma\,(t' - t)\right]$$

p=weight (in brass weights) of 1 cc. of H_2O in vacuo. (This is so nearly 1 that it will not affect the result in the fifth place of decimals, and may therefore be disregarded.) Hence the formula stands:

$$V' = P\frac{1}{d}\left[1 + \gamma\,(t' - t)\right]$$

d=density of water at temperature t.

γ=.000025, the cubical expansion coefficient of glass.

MELTING POINT.

The apparatus for determining the melting point consists of (1) an accurate thermometer for reading easily tenths of a degree; (2) a cathetometer for reading the thermometer (this may be done with an eyeglass if held steadily and properly adjusted); (3) a thermometer; (4) a tall beaker glass 35 cm. high and 10 cm. in diameter; (5) a test tube 30 cm. long and 3.5 cm. in diameter; (6) a stand for supporting the apparatus; (7) some method of stirring the water in the beaker—for example, a blowing bulb of rubber, and a bent glass tube extending to near the bottom of the beaker; (8) a mixture of alcohol and water of the same specific gravity as the fat to be examined.

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* The stopper should be kept for a few minutes before use in hot distilled water.

Manipulation.—The disks of the fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of 15 to 20 cm. on a smooth piece of ice floating in water. The disks thus formed are from 1 to 1.5 cm. in diameter, and weigh about 200 milligrams. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice water before using.

The mixture of alcohol and water is prepared by boiling distilled water and 95 per cent alcohol for ten minutes to remove the gases which they may hold in solution. While still hot the water is poured into the test tube, already described, until it is nearly half full. The test tube is then nearly filled with the hot alcohol. It should be poured in gently down the side of the inclined tube to avoid too much mixing. If the tube is not filled until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top.

The test tube containing the alcohol and water is placed in a tall beaker containing water and ice until cold. The disk of fat is then dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk, the thermometer is moved from time to time in a circularly pendulous manner.

The disk having been placed in position, the water in the beaker glass is slowly heated, and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6° below the melting point, the disk of fat begins to shrivel, and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotatory movement should be given to the thermometer bulb. The rise of temperature should be so regulated that the last 2° of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so, the reading of the thermometer is to be made. As soon as the temperature is taken, the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. The test tube (ice water having been used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum of about 1.5° above the melting point of the fat under examination.

The distilled water for floating the piece of ice on which the disks are made should be recently boiled, to free it of all air particles.

The edge of the disks should not be allowed to touch the sides of the tube. This accident rarely happens, but in case it should take place, and the disk adhere to the sides of the tube, a new trial should be made.

Triplicate determinations should be made, and the second and third results should show a near agreement.

Example.

Melting point of sample of butter:	° C.
First trial	33.15
Second trial	33.05
Third trial	33.00

ESTIMATION OF VOLATILE ACIDS.

Reagents.

(1) *Caustic soda solution.*—100 grams of NaOH are dissolved in 100 cc. of pure water. The caustic soda should be as free as possible from carbonates, and be preserved from contact with the air.

(2) *Alcohol,* of about 95 per cent, redistilled with caustic soda.

(3) *Acid.*—Solution of sulphuric acid containing 25 cc. of strongest H_2SO_4 in 1,000 cc. of water.

(4) *Barium hydrate.*—An accurately standardized, approximately decinormal solution of barium hydrate.

(5) *Indicator.*—Alcoholic solution of phenolphthalein.

Apparatus.

(1) *Saponification flasks* 250 to 300 cc. capacity of hard, well annealed glass capable of resisting the tension of alcohol vapor at 100°.

(2) *A pipette* graduated to deliver 40 cc.

(3) *Distilling apparatus.*

(4) *Burette.*—An accurately calibrated burette reading to tenths of a cubic centimeter.

Manipulation.

Weighing the fat.—The butter or fat to be examined should be melted and kept in a dry warm place at about 60° for two or three hours, until the moisture and curd have entirely settled out. The clean supernatant fat is poured off and filtered through a dry filter paper in a jacketed funnel containing boiling water. Should the filtered fat in a fused state not be perfectly clear, the treatment above mentioned must be repeated.

The saponification flasks are prepared by having them thoroughly washed with water, alcohol, and ether, wiped perfectly dry on the outside, and heated for one hour to boiling temperature. The flasks should then be placed in a tray by the side of the balance and covered with a silk handkerchief until they are perfectly cool. They must not be wiped with a silk handkerchief within fifteen or twenty minutes of the time they are weighed. The weight of the flasks having been accurately determined, they are charged with the melted fat in the following way:

A pipette with a long stem, marked to deliver 5.75 cc., is warmed to a temperature of about 50°. The fat having been poured back and forth once or twice into a dry beaker in order to thoroughly mix it, is taken up in the pipette and the nozzle of the pipette carried to near the bottom of the flask, having been previously wiped to remove any adhering fat, and 5.75 cc. of fat are allowed to flow into the flask. After the flasks have been charged in this way they should be re-covered with the silk handkerchief and allowed to stand fifteen or twenty minutes, when they are again weighed.

The saponification.—Ten cubic centimeters of 95 per cent alcohol are added to the fat in the flask, and then 2 cc. of the concentrated soda solution; a soft cork stopper is now inserted in the flask and tied down with a piece of twine. The saponification is then completed by placing the flask upon the water or steam bath. The flask during the saponification, which should last one hour, should be gently rotated from time to time, being careful not to project the soap for any distance up its sides. At the end of an hour the flask, after having been cooled to near the room temperature, is opened.

Removal of the alcohol.—The stoppers having been laid loosely in the mouth of the flask, the alcohol is removed by dipping the flask into a steam bath. The steam should cover the whole of the flask except the neck. After the alcohol is nearly removed, frothing may be noticed in the soap, and to avoid any loss from this cause or any creeping of the soap up the sides of the flask, it should be removed from the bath and shaken to and fro until the frothing disappears. The last traces of alcohol vapor may be removed from the flask by waving it briskly, mouth down, to and fro.

Dissolving the soap.—After the removal of the alcohol the soap should be dissolved by adding 100 cc. of recently boiled distilled water, warming on the steam bath with occasional shaking until solution of the soap is complete.

Setting free the fatty acids.—When the soap solution has cooled to about 60° or 70° the fatty acids are separated by adding 40 cc. of the dilute sulphuric acid solution mentioned above.

Melting the fatty acid emulsion.—The flask should now be restoppered as in the first instance, and the fatty acid emulsion melted by replacing the flask on the steam bath. According to the nature of the fat examined, the time required for the fusion of the fatty acid emulsions may vary from a few minutes to several hours.

The distillation.—After the fatty acids are completely melted, which can be determined by their forming a transparent oily layer on the surface of the water, the flask is cooled to room temperature, and a few pieces of pumice stone added. The pumice stone is prepared by throwing it, at a white heat, into distilled water, and keeping it under water until used. The flask is now connected with a glass condenser, slowly heated with a naked flame until ebullition begins, and then the distillation continued by regulating the flame in such a way as to collect 110 cc. of the distillate in, as nearly as possible, thirty minutes. The distillate should be received in a flask accurately graduated at 110 cc.

Titration of the volatile acids.—The 110 cc. of distillate, after thorough mixing, are filtered through perfectly dry filter paper and collected in a flask graduated at 100 cc. The 100 cc. of the filtered distillate are poured into a beaker holding from 200 to 250 cc., 0.5 cc. phenolphthalein solution added, and decinormal barium hydrate run in until a red color is produced. The contents of the beaker are then returned to the measuring flask to remove any acid remaining therein, poured again into the beaker, and the titration continued until the red color produced remains apparently unchanged for two or three minutes. The number of cubic centimeters of decinormal $Ba(OH)_2$ required should be increased by one-tenth.

ALTERNATE METHOD OF DETERMINING VOLATILE ACIDS.

Saponification without the use of alcohol.—To avoid the danger of loss from the formation of ethers, and the trouble of removing the alcohol after saponification, the fat may be saponified with a solution of caustic potash in a closed flask without using alcohol. The operation is carried on exactly as indicated above for saponification in a closed flask, using caustic-potash solution instead of soda, and omitting the operation for volatilizing the alcohol. The caustic potash is prepared as follows: Dissolve 100 grams of the purest potassium hydrate in 58 grams of hot distilled water. Allow to cool in a stoppered vessel, decant the clear solution, and preserve in a vessel out of contact with the air. For the saponification use 2 cc. of the caustic-potash solution, which are poured on the fat after it has solidified in the flask. Great care must be taken that none of the fat is allowed to rise on the sides of the saponifying flask to a point where it can not be reached by the alkali. During the process of saponification the flask can only be very gently rotated in order to avoid the difficulty mentioned. This process is not recommended in any except a closed flask with round bottom. In the subsequent solution of the soap use only 80 cc. of distilled water, and in setting free the fatty acids use 60 cc. of the dilute sulphuric acid. In other respects the distillation is conducted as previously described. Potash is used instead of soda, so as to form a softer soap, and thus allow a more perfect saponification.

The saponification may also be conducted as follows: The alkali and fat in the melted state are shaken vigorously in the saponification flask until a complete emulsion is secured. The rest of the operation is then conducted as above.

IODIN ABSORPTION NUMBER.

Reagents.

(1) *Iodin solution.*—Dissolve 25 grams of pure iodin in 500 cc. of 95 per cent alcohol. Dissolve 30 grams of mercuric chlorid in 500 cc. of 95 per cent alcohol. The last solution, if necessary, is filtered, and then the two solutions mixed. The mixed solution should be allowed to stand twelve hours before using.

(2) *Decinormal sodium thiosulphate solution.*—Take 24.6 grams of chemically pure sodium thiosulphate freshly pulverized as finely as possible and dried between filter or blotting paper. Make this up to 1,000 cc. at the temperature at which the titrations are to be made.

(3) *Starch paste.*—One gram of starch boiled in 200 cc. of distilled water for ten minutes and cooled to room temperature.

(4) *Solution of potassium iodid.*—One hundred and fifty grams of potassium iodid dissolved in water and made up to 1 liter.

(5) *Solution of potassium bichromate.*—Dissolve 3.874 grams of chemically pure potassium bichromate in distilled water and make the volume up to 1 liter at the temperature at which the titrations are to made.

Manipulation.

Standardizing the sodium thiosulphate solution.—Run 20 cc. of the potassium bichromate solution, to which has been added 10 cc. of the solution of potassium iodid, into a glass-stoppered flask. Add to this 5 cc. of strong hydrochloric acid. Allow the solution of sodium thiosulphate to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste and with constant shaking continue to add the sodium thiosulphate solution until the blue color just disappears. The number of cubic centimeters of thiosulphate solution used multiplied by 5 is equivalent to 1 gram of iodin.

Example.—Twenty cubic centimeters $K_2Cr_2O_7$ solution required 16.2 cc. sodium thiosulphate; then $16.2 \times 5 = 81 =$ number cubic centimeters of thiosulphate solution equivalent to 1 gram of iodin. Then 1 cc. thiosulphate solution $= 0.0124$ gram of iodin. Theory for decinormal solution of sodium thiosulphate, 1 cc. $= 0.0127$ gram of iodin.

Weighing the sample.—About 1 gram of butter fat is to be weighed in a glass-stoppered flask holding about 300 cc., with the precautions mentioned for weighing the fat for determining volatile acids.

Absorption of iodin.—The fat in the flask is dissolved in 10 cc. of chloroform. After complete solution has taken place, 30 cc. of the iodin-mercuric chlorid solution is added. The flask is now placed in a dark place and allowed to stand, with occasional shaking, for three hours.

Titration of the unabsorbed iodin.—100 cc. of distilled water are added to the contents of the flask, together with 20 cc. of the potassium iodid solution. Any iodin which may be noticed upon the stopper of the flask should be washed back into the flask with the potassium iodid solution. The excess of iodin is now taken up with the sodium thiosulphate solution, which is run in gradually, with constant shaking, until the yellow color of the solution has almost disappeared. A few drops of starch paste are then added, and the titration continued until the blue color has entirely disappeared. Toward the end of the reaction the flask should be stoppered and violently shaken, so that any iodin remaining in solution in the chloroform may be taken up by the potassium iodid solution in the water. A sufficient quantity of sodium thiosulphate solution should be added to prevent a reappearance of any blue color in the flask for five minutes.

Setting the value of the iodin solution by the thiosulphate solution.—At the time of adding the iodin solution to the fats, two blank flasks of the same size as those used for the determination should be employed for conducting the operation described above, but without the presence of any fat. In every other respect the performance of the blank experiments should be just as described. These blank experiments must be made each time the iodin solution is used.

Example—Blank determinations.

(1) 30 cc. iodin solution required 46.4 cc. sodium thiosulphate solution.
(2) 30 cc. iodin solution required 46.8 cc. of sodium thiosulphate solution.
Mean, 46.6.

Per cent of iodin absorbed.

Weight of fat taken	grams	1.0479
Quantity of iodin solution used	cubic centimeters	0.30
Thiosulphate equivalent to iodin used	do	46.6
Thiosulphate equivalent to remaining iodin	do	14.7
Thiosulphate equivalent to iodin absorbed	do	31.9

Per cent of iodin absorbed, $31.9 \times 0.0124 \times 100 \div 1.0479 = 37.75$.

ELECTIVE METHODS OF BUTTER ANALYSIS.

The elective methods of butter analysis remain as described in Bulletin No. 31, pages 200–202.

METHODS FOR MILK ANALYSIS.

DETERMINATION OF WATER.

Evaporate 1 to 2 grams of milk in a tared flat dish containing from 15 to 20 grams of pure dry sand, or without sand, until apparently dry. Dry and heat for one hour at the temperature of boiling water. Cool in a desiccator, and weigh rapidly to avoid absorption of hygroscopic moisture

DETERMINATION OF TOTAL NITROGENOUS MATTER.

Place in a Kjeldahl digestion flask a known weight (about 5 grams) of milk, and proceed, without evaporation, exactly as described for this method under nitrogen.

DETERMINATIONS OF TOTAL SOLIDS AND FAT—BABCOCK ASBESTOS METHOD.

Provide a hollow cylinder of perforated sheet metal, 60 mm. long and 20 mm. in diameter, closed 5 mm. from one end by a disk of the same material. The perforations should be about 0.7 mm. in diameter and about 0.7 mm. apart. Fill loosely with 1.5 to 2.5 grams of freshly ignited woolly asbestos, free from fine and brittle material, cool in a desiccator and weigh. Introduce a weighed quantity of milk (3 to 5 grams) and dry at 100° C. to constant weight for the determination of total solids. Extract with anhydrous ether until fat is removed, evaporate the ether, dry the fat at 100° C. and weigh. The fat may also be determined by difference, drying the extracted cylinders at 100° C.

DETERMINATION OF FAT—PAPER-COIL METHOD.

Coils made of thick filter paper, cut into strips 6.25 by 62.5 mm. are thoroughly extracted with ether and alcohol, or the weight of the extract corrected by a constant obtained for the paper. If this latter method is used a small amount of anhydrous sodium carbonate should be added. From a weighing bottle about 5 grams of milk are transferred to the coil by a pipette, care being taken to keep the end of the coil held in the fingers dry. The coil, dry end down, on a piece of glass, is dried at the temperature of boiling water for one hour, or, better, dried in hydrogen at the temperature of boiling water, transferred to an extraction apparatus, and extracted with absolute ether or petroleum spirit boiling at about 45°. The extracted fat is dried in hydrogen and weighed.

If the milk is sour add about 10 per cent, by weight, of strong ammonia water, and a small quantity of anhydrous sodium carbonate, correcting the results for the ammonia added.

DETERMINATION OF SUGAR.

Reagents.

(1) *Acid mercuric nitrate.*—Dissolve mercury in double its weight of nitric acid, sp. gr. 1.42. Add to the solution an equal volume of water. One cubic centimeter of this reagent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting the results of polarization.

(2) *Mercuric iodid with acetic acid.*—KI 33.2 grams, $HgCl_2$ 13.5 grams, $C_2H_4O_2$ 20 cc., H_2O 640 cc.

Apparatus.

(1) Pipettes marked at 59.5, 60, and 60.5 cc. (2) Sugar flasks marked at 102.4 cc., (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

Manipulation.

(1) The room and milk should be kept at a constant temperature.

(2) The specific gravity of the milk is determined. For general work this is done by a delicate specific gravity spindle. Where greater accuracy is required use specific gravity flasks.

(3) If the specific gravity be 1.026, or nearly so, place 60.5 cc. in the sugar flask. Add 1 cc. of mercuric nitrate solution, or 30 cc. of mercuric iodid solution, and fill to the 102.4 cc. mark. The precipitated albumen occupies a volume of about 2.44 cc. Hence the milk solution is really 100 cc. If the specific gravity is 1.030, use 60 cc. of milk. If the specific gravity is 1.034, use 59.5 cc. of milk.

(4) Fill up to the mark in the 102.4 cc. flask, shake well, filter, and polarize.

NOTES.—In the above method of analysis the specific rotary power of milk sugar is taken at 52.5 and the weight of it in 100 cc. solution to read 100° on the cane sugar scale at 20.56 grams. This is for instruments requiring 16.19 grams sucrose to produce a rotation of 100° sugar. It will be easy to calculate the number for milk sugar, whatever instrument is employed.

Since the quantity of milk taken is three times 20.56 grams, the polariscopic readings divided by 3 give at once the percentage of milk sugar when a 200-mm. tube is used.

If a 400-mm. tube is employed, divide the reading by 6; if a 500-mm. tube is used, divide by 7.5.

By using a flask graduated at 102.4 cc. for 60 cc. no correction for volume of precipitated casein need be made. In no case is it necessary to heat the sample before polarizing.

ALTERNATE METHOD FOR SUGAR.

The sugar may also be determined, either gravimetrically or volumetrically, by alkaline copper solution, as described under sugar analysis.

DETERMINATION OF ASH.

In a weighed dish put 20 cc. of milk from a weighing bottle; add 6 cc. of HNO_3, evaporate to dryness, and burn at low red heat until ash is free from carbon.

PROVISIONAL METHODS OF CHEESE ANALYSIS.

SAMPLING.

Where the cheese can be cut, a narrow wedge reaching from the edge to the center of the cheese will more nearly represent the average composition of the cheese than any other sample. This may be chopped quite fine, with care to avoid evaporation of water, and the several portions for analysis taken from the mixed mass, when the sample is taken with a cheese trier, a plug taken perpendicular to the surface one-third of the distance from the edge to the center of the cheese should more nearly represent the average composition than any other. The plug should either reach entirely through or only half way through the cheese. For inspection purposes the rind may be rejected, but for investigations, where the absolute quantity of fat in the cheese is required, the rind should be included in the sample. It is well, when admissible, to take two or three plugs on different sides of the cheese, and, after splitting them lengthwise with a sharp knife, take portions of each for the different determinations.

DETERMINATION OF WATER.

From 5 to 10 grams of cheese should be taken, and placed in thin slices in a weighed platinum or porcelain dish which contains a small quantity of freshly ignited asbestus, to absorb the fat which may run out of the cheese. This is then heated in a boiling water oven for ten hours and weighed; the loss in weight shall be considered as water. Or, if preferred, the dish may be placed in a desiccator over concentrated sulphuric acid and dried to constant weight. In some cases this may require as much as two months. The acid should be renewed when the cheese has become nearly dry.

DETERMINATION OF ASH.

The dry residue from the water determination may be taken for the ash. If the cheese be rich in fat the asbestos will be saturated therewith. This may be ignited carefully and the fat allowed to burn off, the asbestos acting as a wick. No extra heat should be applied during this operation, as there is danger of spurting. When the flame has died out the burning may be completed in a muffle at low redness. When desired, the salt may be determined in the ash in the manner specified for butter.

DETERMINATION OF FAT.

Take 5 to 10 grams of cheese and grind it in a small mortar with about twice its weight of anhydrous copper sulphate. The grinding should continue until the cheese is finely pulverized and evenly distributed throughout the mass, which will have a uniform light blue color. This mixture is transferred to a glass tube which has a strong filter paper, supported by a piece of muslin, tied over one end. Put a little of the clean anhydrous copper sulphate into the tube next to the filter before introducing the mixture containing the cheese. On top of the mixture place a tuft of ignited asbestos, and place the tube in a continuous extraction apparatus and extract with anhydrous ether for fifteen hours. Dry the fat obtained at 100° to constant weight.

DETERMINATION OF CASEIN.

Make a determination of nitrogen by the Kjeldahl method, taking about 2 grams of cheese, and multiply the result by 6.25 for casein.

OTHER CONSTITUENTS.

The sum of the percentages of the different constituents, determined as above, subtracted from 100 will give the amount of organic acids, milk sugar, etc., in the cheese.

METHODS OF ANALYSIS FOR FERMENTED LIQUORS.

I. *Specific gravity.*—The specific gravity is taken at 15.5° by means of a pyknometer or a Westphal balance controlled by a pyknometer; or a delicate spindle may be used which has been carefully calibrated by a pyknometer.

II. (*a*) *Alcohol by weight.*—About 100 grams of the liquor are weighed or 100 grams measured from a burette at 15.5°, as determined by the specific gravity, 50 cc. of water added, and 100 cc. distilled in an appropriate distilling apparatus provided with a safety bulb and mercury valve. The specific gravity of the distillate is determined as in I. It is also weighed or its weight calculated from the specific gravity. The corresponding percentage of alcohol is obtained from the official tables (p. 381), and this figure multiplied by the weight of the distillate and the result divided by the weight of the sample taken gives the per cent of alcohol by weight contained therein.

(*b*) *Alcohol by volume.*—The percentage of alcohol by volume of the liquor is the same as that of the distillate and is obtained from the official tables (p. 381).

III. *Extract.*—(*a*) Dry wines, beers, ales, etc. Fifty cubic centimeters are weighed in a platinum dish 85 mm. in diameter and capable of holding 75 cc. and evaporated on the water bath to a sirupy consistence. The residue is heated for two and a half hours in a drying oven at the temperature of boiling water.

(*b*.) Sweet wines. (1) Of liquors containing more than 4 per cent solids not more than 10 cc. should be taken.

(2) Ten cubic centimeters of the liquor are weighed and diluted to 100 cc. with water. Fifty cubic centimeters of this solution are evaporated as described under (*a*).

(3) Alternate method. Fifty cubic centimeters of wine are carefully measured into a platinum or porcelain dish and evaporated on the water bath until the volume is reduced to one-third. The dealcoholized liquid is washed into a 50 cc. flask, cooled, and made up to the original volume. It is mixed thoroughly and the specific gravity ascertained with a pyknometer or an accurately standardized spindle. The per cent of total solids, in case the pyknometer is used, is obtained from the appended table.

IV. *Total acidity* (*expressed as tartaric acid*).—Expel any carbonic acid that is present by continued shaking. Transfer 10 cc. to a beaker and, in the case of white wines, add about 10 drops of a neutral litmus solution. Add $\frac{N}{10}$ sodium hydrate solution until the red color changes to violet. Then add the reagent, a few drops at a time until a drop of the liquid, placed on delicate red litmus paper, shows an alkaline reaction.

One cubic centimeter $\frac{N}{10}$ sodium hydrate solution = 0.0075 gram tartaric acid.

V. *Volatile acids* (*expressed as acetic acid*).—Fifty cubic centimeters of wine, to which a little tannin has been added to prevent foaming, are heated in a flask which is connected with a flask supplying steam and a vertical condenser. Both flasks are heated until the liquids are boiling, when the lamp under the flask containing the wine is turned down, and the steam passed through until 200 cc. have been collected in the receiver. The distillate is titrated with $\frac{N}{10}$ sodium hydrate solution and computed as acetic acid.

One cubic centimeter $\frac{N}{10}$ sodium hydrate solution = 0.0060 gram acetic acid.

VI. *Glycerol.*—(*a*) In dry wines. One hundred cubic centimeters of wine are evaporated in a porcelain dish to about 10 cc., a little quartz sand and milk of lime added, and the evaporation carried almost to dryness. The residue is mixed with 50 cc. of 90 per cent alcohol, using a glass pestle or spatula to break up any solid particles. It is then heated to boiling on the water bath, allowed to settle, and the liquid filtered into a small flask. The residue is repeatedly extracted in a similar manner with small portions of boiling alcohol until the filtrate in the flask amounts to about 150 cc. A little quartz sand is then added to it, the flask connected with a condenser, and the alcohol distilled slowly until about 10 cc. remain.

The evaporation is then continued on the water bath until the residue becomes sirupy. It is cooled and dissolved in 10 cc. of absolute alcohol. Solution may be facilitated by gentle heating on the water bath. Fifteen cubic centimeters of anhydrous ether are then added and the flask stoppered and allowed to stand until the precipitate has collected on the sides and bottom of the flask. The clear liquid is decanted into a tared weighing bottle, the precipitate repeatedly washed with a few cubic centimeters of a mixture of 1 part absolute alcohol and 1.5 parts anhydrous ether and the washings added to the solution. The ether-alcohol is removed on the water bath and the residue dried one hour in a water oven.

(*b*) In sweet wine. One hundred cubic centimeters of wine are evaporated on the water bath to a sirupy consistence, a little quartz sand being added to render subsequent extraction easier. The residue is repeatedly extracted with absolute alcohol until the united extracts amount to 100 to 150 cc. The extract is collected in a flask and for every part of alcohol used 1.5 parts of ether are added, the liquid well shaken, and allowed to stand until it becomes clear. The supernatant liquid is decanted into a beaker, and the precipitate washed with a few cubic centimeters of a mixture of one part alcohol and 1½ parts ether. The united liquids are distilled, the evaporation being finished on the water bath, the residue is dissolved in water, transferred to a porcelain dish, and treated as under (*a*).

(*c*) It is necessary to test the glycerol from sweet wines for sugar, and if any is present it must be estimated by Fehling-Soxhlet's method, given below, and its weight subtracted from that of the glycerol.

VII. *Reducing sugar.*—Fehling-Soxhlet method. For reagents required see methods for sugar analysis. In applying this method it is necessary to use solutions containing not more than 1 per cent of sugar.

(1) Preliminary test. One hundred cubic centimeters of wine are accurately neutralized with normal sodium hydrate solution, dealcoholized by evaporation to one-third, cooled, and the original volume restored with water. Red wines must be decolorized with animal charcoal. Twenty-five cubic centimeters of copper solution and 25 cc. of Seignette salt solution are heated to boiling in a deep porcelain evaporating dish. The wine is then added from a burette until the fluid, after two minutes' boiling, no longer has a blue color. This gives an approximate estimate of the amount of reducing sugar present, and the wine is then diluted so as to contain about 1 per cent.

(2) Final determination. Twenty-five cubic centimeters of the copper solution, 25 cc. of the Seignette salt solution, and 23 to 24 cc. of the diluted wine are heated two minutes to boiling, the precipitated copper oxid allowed to settle and a small quantity of the liquid filtered through a folded filter. If the filtrate is greenish it contains copper; if it is yellow it is cooled, acidulated with acetic acid, and tested with a few drops of potassium ferrocyanid solution. A reddish color indicates copper, and shows that not enough wine has been added. The experiment is repeated, adding 1 cc. more if copper was present, or 1 cc. less if it was not present. The experiments are repeated until filtrates are obtained representing a difference of 0.1 cc. of wine, one of which contains copper and one not. The mean of the two contains the amount of sugar equivalent to 50 cc. of Fehling solution.

Fifty cubic centimeters Fehling solution = 0.2375 gram grape sugar.

(3) Allihn's method. (See this method under sugar analysis.)

VIII. *Polarization.*—Whenever practicable angular rotation (Wild polaristrobometer) is to be used. When any other instrument is used the results are to be calculated to equivalents of Wild's polaristrobometer (angular rotation) in a 220 mm. tube. All results are to be stated as the polarization of the undiluted wine.

Factors: 1° Wild = 2.89005 Ventzke-Soleil or Schmidt and Haensch.
1° Ventzke-Soleil or Schmidt and Haensch = 0.346015° Wild.

(*a*) In white wines. Sixty cubic centimeters of wine are decolorized with 3 cc. of lead subacetate and filtered. To 30 cc. of the filtrate 1½ cc. of a saturated solution of sodium carbonate are added, filtered and the filtrate polarized. This gives a dilution of 10 to 11 which must be considered in the calculation.

(*b*) In red wines. Sixty cubic centimeters of wine are decolorized with 6 cc. of lead subacetate and filtered. To 30 cc. of the filtrate 3 cc. of a saturated solution of sodium carbonate are added, filtered and the filtrate polarized. The dilution in this case is 5 to 6.

(*c*) In sweet wines.

(1) Before inversion. Twenty-five cubic centimeters are decolorized in a 50 cc. flask with 1 cc. of lead subacetate, one-half cubic centimeter of a saturated solution of sodium carbonate is added, the liquid made up to the mark with water, filtered and polarized. The dilution is 1 to 2.

(2) The wine is polarized after inversion. Twenty-five cubic centimeters of wine are placed in a 50 cc. flask with 2.5 cc. strong hydrochloric acid. After mixing well the flask is placed in water and heated until a thermometer, placed in the flask with the bulb as near the center of the liquid as possible, marks 68°, consuming about ten minutes in the heating. It is then removed, cooled quickly to room temperature, decolorized with lead subacetate and sodium carbonate, made up to the mark, filtered and polarized, the temperature being noted.

(3) After fermentation. Fifty cubic centimeters of wine, which has been dealcoholized and made up to the original volume with water, are mixed in a small flask with well-washed beer yeast and kept at 30° C. until fermentation has ceased, which requires from two to three days. The liquid is then washed into a 100 cc. flask, a few drops of a solution of acid mercuric nitrate and then lead subacetate and sodium carbonate solution added. The flask is filled to the mark with water, shaken, the solution filtered and polarized.

Application of the analytical methods.—(*a*) The wine shows no rotation. This may be due to the absence of any rotatory body; to the simultaneous presence of the dextrorotatory nonfermentable constituents of commercial dextrose and levorotatory sugar, or to the simultaneous presence of dextrorotatory cane sugar and levorotatory invert sugar.

(1) The wine is inverted. A levorotation shows the presence of cane sugar.

(2) The wine is fermented. A dextrorotation shows that both levorotatory sugar and the unfermentable constituents of commercial dextrose are present.

If in both cases no change took place it proves the absence of unfermented cane sugar, the unfermentable constituents of commercial dextrose and of levorotatory sugar.

(*b*) The wine is dextrorotatory. This may be caused by unfermented cane sugar, the unfermentable constituents of commercial dextrose, or both.

(1) The wine is inverted.

(α) It rotates to the left after inversion. Unfermented cane sugar is present.

(β) It rotates more than 0.8° Wild to the right. The unfermentable constituents of commercial dextrose are present.

(γ) It rotates less than 0.8° and more than 0.3° Wild to the right. It is in this case treated as follows:

Two hundred and ten cubic centimeters of the wine are evaporated in a porcelain dish to a thin sirup with a few drops of a 20 per cent solution of potassium acetate. To the residue 200 cc. of 90 per cent alcohol are added with constant stirring. The alcoholic solution is filtered into a flask, and the alcohol removed by distillation until about 5 cc. remain. The residue is mixed with washed boneblack, filtered into a graduated cylinder, and washed until the filtrate amounts to 30 cc. When the filtrate shows a dextrorotation of more than 0.5° Wild it proves the presence of the unfermentable constituents of commercial dextrose.

(*c*) The wine rotates to the left. It contains unfermented levorotatory sugar, derived either from the must or from the inversion of added cane sugar. It may, however, also contain unfermented cane sugar and the unfermentable constituents of commercial dextrose.

(1) The wine is fermented according to VIII (c), 3.

(α) It polarizes 10° after fermentation. It contains only levorotatory sugar.

(β) It rotates to the right. It contains both levorotatory sugar and the unfermentable constituents of commercial dextrose.

(2) The wine is inverted according to VIII (c), 2.

(α) It is more strongly levorotatory after inversion. It contains both levorotatory sugar and unfermented cane sugar.

IX. *Tannin and coloring matter.*—Determine by the Löwenthal-Neubauer method. Reagents: Potassium permanganate solution, 1.333 grams $KMnO_4$ are dissolved in 1 liter of water.

Indigo solution: 6 grams of sodium indigo sulphate are dissolved in 500 cc. of water, with the aid of heat cooled, 50 cc. of concentrated sulphuric acid added and the solution made up to 1 liter and filtered.

$\frac{N}{10}$ solution of oxalic acid: 10 cc. = 0.04157 gram tannin.

Purified boneblack: Finely pulverized boneblack is extracted with hydrochloric acid, and washed with distilled water until all the acid has been removed. The boneblack is kept just covered with water.

The potassium permanganate solution is standardized by means of the $\frac{N}{10}$ oxalic acid solution.

(1) One hundred cubic centimeters of wine are weighed in a flask, dealcoholized and the original volume restored with water. Ten cubic centimeters are measured by means of a burette in a porcelain casserole, having a capacity of about a liter and 750 cc. of water and 20 cc. of the indigo solution added, the latter being measured from a burette. The potassum permanganate solution is then added from a burette, a cubic centimeter at a time, until the blue color changes to green, then a few drops at a time until the liquid becomes golden yellow. Result a.

(2) Ten cubic centimeters of the dealcoholized wine, measured out as in 1, are treated with boneblack for fifteen minutes, filtered, and the boneblack washed carefully. The filtrate is diluted with 750 cc. of water, 20 cc. of indigo solution added, and the titration carried out as in 1. Result b.

Result a — Result b = Result c = the number of cubic centimeters of the potassium permanganate solution required for the oxidation of the tannin and coloring matter in 10 cc. of wine.

X. *Potassium bitartrate.*—The determination of potassium bitartrate is necessary when an estimation of the tartaric acid is desired.

Fifty cubic centimeters of wine are measured into a porcelain dish and evaporated to a sirupy consistence, a little quartz sand being added to render subsequent extraction easier. After cooling, 70 cc. of 96 per cent alcohol are added with constant stirring. After standing for twelve hours at as low a temperature as practicable, the solution is filtered off and the precipitate washed with alcohol until the filtrate is no longer acid. The alcoholic filtrate is preserved for the estimation of the tartaric acid. The filter and precipitate are returned to the porcelain dish and repeatedly treated with hot water, each extraction being filtered into a flask or beaker until the washings are neutral. The combined filtrates and washings are then titrated with $\frac{N}{10}$ sodium hydrate solution.

One cubic centimeter $\frac{N}{10}$ sodium hydrate solution = 0.0188 potassium bitartrate.

XI. *Tartaric, malic, and succinic acids.*—(a) According to Schmidt and Hiepe's method.

Two hundred cubic centimeters of wine are evaporated one-half, cooled, and basic lead acetate added until the reaction is alkaline. The precipitate is filtered off and washed with cold water until the filtrate shows only a slight reaction for lead. The precipitate is washed from the filter into a beaker by means of hot water, and treated hot with H_2S until all the lead is converted into sulphid. It is then filtered hot and the lead sulphid washed with hot water until the washings are no longer acid. The filtrate and washings are evaporated down to 50 cc. and accurately neutralized with potassium hydrate. An excess of a saturated solution of calcium acetate is added and the liquid allowed to stand from four to six hours with frequent stirring. It is then filtered and the precipitate washed until the filtrate amounts to exactly 100 cc. The precipitate of calcium tartrate is converted into calcium oxid by igniting in a platinum crucible. After cooling, from 10 to 15 cc. of normal hydrochloric acid are added, the solution washed into a beaker and accurately titrated with normal potassium hydrate solution. Every cubic centimeter of normal acid saturated by the calcium oxid is equivalent to 0.075 gram tartaric acid. To the amount so obtained, 0.0286 gram must be added representing the tartaric acid held in solution in the filtrate as calcium tartrate. The sum represents the total tartaric acid in the wine.

The filtrate from the calcium tartrate is now evaporated to about 20 to 30 cc., cooled, and mixed with three times its volume of 96 per cent alcohol. After standing several hours the precipitate is collected on a weighed filter, dried at 100° C., and weighed. It represents the calcium salts of malic, succinic, and sulphuric acids and of the tartaric acid which remained in solution. This precipitate is dissolved in a minimum quantity of hydrochloric acid, filtered, and the filter washed with hot water. Potassium carbonate solution is added to the hot filtrate and the precipitated calcium carbonate filtered off and washed. This filtrate contains the potassium salts of the above-named acids. It is neutralized with acetic acid, evaporated to a small volume, and precipitated hot with $BaCl_2$. The precipitate of barium succinate and sulphate is filtered off, washed with hot water, and treated on the filter with dilute hydrochloric acid. The $BaSO_4$ remaining behind is washed, dried, ignited, and weighed. In the filtrate which contains the succinic acid and the barium equivalent to it, the latter is precipitated hot with sulphuric acid, washed, dried, ignited, and weighed. Two hundred and twenty-three parts $BaSO_4$ equal 118 parts succinic acid. The succinic and sulphuric acids, as well as the tartaric acid remaining in solution, which was equal to 0.0286 gram, are to be calculated as calcium salts and the result deducted from the total weight of the calcium precipitate. The remainder is the calcium malate, of which 172 parts equal 134 parts malic acid.

(*b*) Determination of the tartaric acid according to the modified Berthelot-Fleury method.

Fifty cubic centimeters of wine are measured, and 10 cc. of this neutralized with potassium hydrate solution. The two liquids are then mixed, and to one-fifth of the volume, corresponding to 10 cc. of wine, 50 cc. of a mixture of equal parts of alcohol and ether are added and the mixture allowed to stand twenty-four hours. The precipitated calcium bitartrate is filtered off, dissolved in water, and titrated. The excess of potassium bitartrate over the amount of that constituent present in the wine corresponds to the free tartaric acid.

(*c*) Determination of the tartaric acid in the filtrate from the potassium bitartrate X.

The filtrate is made up to a definite volume with water and divided into two equal portions. One portion is exactly neutralized with $\frac{N}{10}$ sodium hydrate solution, the other portion added, the alcohol distilled off, the residue washed into a porcelain dish, and treated as under X.

One cubic centimeter $\frac{N}{10}$ sodium hydrate solution = 0.0075 gram tartaric acid. As, however, only half of the free tartaric acid is determined by this method 1 cc. $\frac{N}{10}$ sodium hydrate = 0.0150 gram of the tartaric acid contained in the wine.

XII. *Coloring matter.* (*Only anilin dyes are to be looked for.*)

(*a*) Fuchsin. To 20 cc. of wine add 10 cc. of lead acetate solution, heat slightly, and mix by shaking. Filter into a test tube, add 2 cc. of amyl alcohol, and shake. If the amyl alcohol is colored red, separate it and divide it into two portions. To one add hydrochloric acid, to the other ammonia. When the color is due to fuchsin the amyl alcohol will in both cases be decolorized; when due to orseille the ammonia will change the color of the amyl alcohol to purple-violet.

(*b*) Sodium rosanilinsulphonate. Method of R. Kayser. The wine is acidified with acetic acid and shaken up with amyl alcohol. The amyl alcohol is examined spectroscopically for the rosanilin spectrum.

XIII. *Inorganic matter (ash).*—The residue from the direct extract determination is incinerated at as low a heat as possible. Repeated moistening, drying, and heating to redness is advisable to get rid of all organic substances.

XIV. *Citric acid.*—Presence to be shown by a qualitative test, as barium citrate.

XV. *Sulphuric acid.*—Fifty cubic centimeters of wine are acidified with hydrochloric acid and precipitated with barium chlorid.

XVI. *Chlorin.*—To be determined in the nitric-acid solution of the burned residue.

XVII. *Calcium and magnesium.*—Fifty cubic centimeters of wine are evaporated to dryness and the residue incinerated. The ash is dissolved in hydrochloric acid and filtered into a beaker. To the filtrate ammonia is added until the reaction is strongly alkaline, and then acetic acid until the precipitate caused by the ammonia is almost all dissolved and the liquid is acid. The precipitate of iron and aluminum phosphates, which is usually very small, is filtered off and washed. The filtrate is heated to boiling and ammonium oxalate added until all the calcium is precipitated. The calcium oxalate is collected on a Gooch crucible, washed with hot water, dried, and either weighed as calcium oxalate or ignited and weighed as oxid.

Magnesium.—The filtrate and washings from the calcium oxalate are evaporated in a beaker to about 10 cc. and cooled. Three cubic centimeters of sodium phosphate solution are then added, with constant stirring, and after about fifteen minutes 4 cc. of ammonia. After standing for twelve hours the precipitate is filtered off and treated as usual.

XVIII. *Phosphoric acid.*—The ash from 50 cc. of wine is treated with nitric acid and hot water. The solution is filtered into a beaker of about 150 cc. capacity. The phosphoric acid is then determined by the molybdate method.

XIX. *Potash.*

(*a*) Determined by Kayser's method. 0.7 gram crystallized sodium hydrate and 2 grams tartaric acid are dissolved in 100 cc. of wine, 150 cc. of 92 to 94 per cent alcohol added, and the liquid allowed to stand twenty-four hours. The precipitated potassium bitartrate is collected on a small filter and washed with 50 per cent alcohol until the filtrate amounts to 260 cc. The precipitate and filter are then transferred to the beaker in which the precipitation was made, dissolved in hot water, made up to 200 cc. and 50 cc., titrated with $\frac{N}{10}$ sodium hydrate solution; 0.004 gram must be added to the final result, this representing the potash which remains in solution as bitartrate.

(*b*) In the ash of 100 cc. of wine as potassium-platinum chlorid.

XX. *Gum and dextrin.*—Four cubic centimeters of wine are mixed with 10 cc. of 96 per cent alcohol. When gum arabic or dextrin is present a lumpy, thick, and stringy precipitate is produced, whereas pure wine becomes at first opalescent and then gives a flocculent precipitate.

XXI. *Sulphurous acid.*—One hundred cubic centimeters of wine are distilled in a current of carbonic-acid gas after the addition of phosphoric acid until about 50 cc. have passed over. The distillate is collected in accurately standardized iodin solution. When the distillation is finished the excess of iodin is determined with standardized sodium thiosulphate solution, and the sulphurous acid calculated from the iodin used.

XXII. *Salicylic acid.*—Method of W. D. Bigelow. Place 100 cc. of the wine in a separatory funnel, add 5 cc. of sulphuric acid (1:3) and extract with a sufficient quantity of a mixture of 8 to 9 parts of ether to 1 part of petroleum ether. Throw away the aqueous solution, wash the ether once with water, then shake thoroughly with about 50 cc. of water, to which from 6 to 8 drops of 0.5 per cent solution of Fe_2Cl_6 have been added. Discard the aqueous solution, wash again with water, transfer the ethereal solution to a porcelain dish, and allow to evaporate spontaneously.

Then heat the dish on the steam bath, take up with 1 to 2 cc. of cold water, filter into a test tube, and add 1 to 2 drops of 0.5 per cent solution of Fe_2Cl_6. The presence of salicylic acid is indicated by the appearance of a violet-red coloration. In the case of red wines a second extraction of the residue with ether mixture is sometimes necessary. This is determined by the amount of residue left in the dish on the evaporation of the ether.

Hager-Schulze table for the determination of the extract by the indirect method.

Hager.	Specific gravity.	Schulze	Hager.	Specific gravity.	Schulze	Hager.	Specific gravity.	Schulze	Hager.	Specific gravity.	Schulze
0.84	1.0038	1.00	1.39	1.0063	1.64	1.94	1.0088	2.28	2.48	1.0113	2.92
0.86	1.0039	1.02	1.42	1.0064	1.67	1.96	1.0089	2.30	2.50	1.0114	2.94
0.88	1.0040	1.05	1.44	1.0065	1.69	1.98	1.0090	2.33	2.52	1.0115	2.97
0.90	1.0041	1.08	1.46	1.0066	1.72	2.00	1.0091	2.35	2.54	1.0116	2.99
0.92	1.0042	1.10	1.48	1.0067	1.74	2.03	1.0092	2.38	2.57	1.0117	3.02
0.94	1.0043	1.13	1.50	1.0068	1.77	2.05	1,0093	2.41	2.59	1.0118	3.05
0.96	1.0044	1.15	1.52	1.0069	1.79	2 07	1.0094	2.43	2.61	1.0119	3.07
0.98	1.0045	1.18	1.55	1.0070	1.82	2.09	1.0095	2.46	2.64	1.0120	3.10
1.00	1 0046	1.21	1.57	1.0071	1.84	2.11	1.0096	2.48	2.66	1.0121	3.12
1.02	1.0047	1.23	1.59	1.0072	1.87	2.14	1.0097	2.51	2.68	1.0122	3.15
1.04	1.0048	1.26	1.61	1.0073	1.90	2.16	1.0098	2.53	2.70	1.0123	3.17
1.06	1.0049	1.29	1.64	1.0074	1.92	2.18	1.0099	2.56	2.72	1.0124	3.20
1.08	1.0050	1.31	1.66	1.0075	1.95	2.21	1.0100	2.58	2.75	1.0125	3.23
1.10	1.0051	1.34	1.68	1.0076	1.97	2.23	1.0101	2.61	2.77	1.0126	3.25
1.12	1.0052	1.36	1.70	1.0077	2.00	2.25	1.0102	2.64	2.79	1.0127	3.28
1.15	1.0053	1.39	1.72	1.0078	2.02	2.27	1.0103	2.66	2.82	1.0128	3.30
1.17	1.0054	1.41	1.75	1.0079	2.05	2.30	1.0104	2.69	2.84	1.0129	3.33
1.19	1.0055	1.44	1.77	1.0080	2.07	2.32	1.0105	2.71	2.86	1.0130	3.35
1.22	1.0056	1.46	1.79	1.0081	2.10	2.34	1.0106	2.74	2.88	1.0131	3.38
1.25	1.0057	1.49	1.82	1.0082	2.12	2.36	1.0107	2.76	2.90	1.0132	3.41
1.27	1.0058	1.51	1.84	1.0083	2.15	2.38	1.0108	2.79	2.92	1.0133	3.43
1.30	1.0059	1.54	1.86	1.0084	2.17	2.40	1.0109	2.82	2.94	1.0134	3.46
1.32	1.0060	1.56	1.88	1.0085	2.20	2.42	1.0110	2.84	2.96	1.0135	3.48
1.34	1.0061	1.59	1.90	1.0086	2.23	2.44	1.0111	2.87	2.98	1.0136	3.51
1.37	1.0062	1.62	1.92	1.0087	2.25	2.46	1.0112	2.89	3.00	1.0137	3.54

TABLE I.—*Correction to be applied to the reading of Balling's saccharometer for different temperatures.*

	Temp., ° F.	Degree Balling.																				
		1°	2°	3°	4°	5°	6°	7°	8°	9°	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°	21°
To be subtracted from the indicated degree.	50	0.17	0.17	0.18	0.19	0.20	0.20	0.21	0.21	0.22	0.22	0.22	0.23	0.23	0.24	0.24	0.24	0.25	0.25	0.25	0.26	0.26
	51	.16	.16	.17	.18	.19	.19	.20	.20	.21	.21	.21	.22	.22	.23	.23	.23	.24	.24	.24	.24	.25
	52	.15	.15	.16	.17	.18	.18	.18	.19	.19	.19	.19	.19	.20	.20	.21	.21	.22	.22	.22	.22	.23
	53	.14	.14	.14	.15	.16	.16	.16	.17	.17	.18	.18	.18	.18	.19	.19	.19	.19	.20	.20	.20	.20
	54	.12	.12	.13	.14	.15	.15	.15	.16	.16	.16	.16	.17	.17	.17	.17	.17	.18	.18	.18	.18	.18
	55	.09	.09	.09	.10	.11	.11	.11	.12	.12	.12	.12	.13	.13	.13	.14	.14	.14	.14	.14	.14	.15
	56	.07	.07	.07	.08	.09	.09	.09	.09	.10	.10	.10	.10	.11	.11	.11	.11	.11	.11	.11	.11	.11
	57	.06	.06	.06	.07	.07	.07	.07	.07	.08	.08	.08	.08	.08	.09	.09	.09	.09	.10	.10	.10	.10
	58	.04	.04	.04	.05	.05	.05	.05	.05	.06	.06	.06	.06	.06	.07	.07	.07	.07	.07	.07	.07	.07
	59	.02	.02	.02	.02	.02	.02	.02	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03
	60																					
To be added to the indicated degree.	61	.02	.02	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03
	62	.06	.06	.07	.07	.08	.08	.08	.08	.08	.08	.09	.09	.09	.09	.09	.09	.09	.09	.09	.09	.09
	63	.09	.10	.10	.11	.11	.11	.11	.11	.11	.11	.12	.12	.12	.12	.12	.12	.12	.12	.13	.12	.12
	64	.12	.12	.13	.13	.14	.14	.14	.14	.15	.15	.15	.15	.16	.16	.17	.17	.17	.17	.17	.17	.17
	65	.14	.15	.15	.16	.17	.17	.17	.17	.18	.18	.18	.19	.19	.19	.20	.20	.20	.20	.20	.20	.20
	66	.17	.17	.18	.19	.20	.20	.21	.21	.21	.22	.22	.23	.23	.23	.24	.24	.24	.24	.24	.24	.24
	67	.20	.21	.21	.22	.23	.23	.23	.24	.24	.25	.25	.25	.26	.26	.27	.27	.27	.27	.27	.27	.27
	68	.22	.23	.24	.25	.26	.26	.27	.28	.28	.29	.29	.30	.30	.31	.31	.31	.31	.31	.31	.31	.31
	69	.25	.26	.27	.28	.29	.29	.30	.31	.31	.32	.32	.33	.33	.34	.34	.34	.34	.34	.34	.34	.34
	70	.28	.29	.30	.31	.32	.32	.33	.34	.34	.35	.35	.36	.36	.37	.37	.37	.37	.38	.38	.38	.38
	71	.31	.32	.33	.34	.35	.35	.36	.37	.37	.38	.38	.39	.39	.40	.40	.40	.40	.40	.40	.40	.41
	72	.34	.35	.36	.37	.38	.39	.40	.41	.42	.42	.42	.43	.43	.43	.44	.44	.44	.44	.44	.44	.44
	73	.37	.38	.40	.41	.42	.43	.44	.44	.45	.46	.46	.46	.46	.47	.47	.47	.47	.47	.47	.47	.47
	74	.40	.42	.43	.45	.46	.47	.48	.48	.49	.50	.50	.50	.51	.51	.51	.51	.51	.52	.52	.52	.52
	75	.44	.46	.47	.49	.50	.51	.52	.52	.53	.54	.54	.54	.55	.55	.56	.56	.56	.57	.57	.58	.58
	76	.47	.48	.50	.52	.53	.54	.55	.55	.56	.57	.57	.58	.58	.59	.60	.60	.61	.61	.61	.62	.62
	77	.50	.52	.54	.56	.57	.58	.59	.60	.60	.61	.61	.62	.62	.63	.63	.63	.64	.64	.64	.65	.65
	78	.53	.54	.56	.58	.60	.61	.62	.62	.63	.64	.64	.65	.65	.66	.66	.66	.67	.67	.68	.68	.68
	79	.57	.58	.60	.62	.64	.65	.66	.66	.67	.68	.68	.69	.69	.70	.70	.70	.71	.71	.71	.72	.72
	80	.60	.62	.64	.66	.68	.69	.70	.70	.71	.72	.72	.72	.73	.73	.74	.74	.75	.75	.75	.76	.76

TABLE I.—*Correction to be applied to the reading of Balling's saccharometer for different temperatures*—Continued.

	Temp., ° F.	Degree Balling.																				
		1°	2°	3°	4°	5°	6°	7°	8°	9°	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°	21°
To be added to the indicated degree.	81	0.63	0.65	0.67	0.69	0.71	0.72	0.73	0.73	0.74	0.75	0.75	0.76	0.76	0.77	0.78	0.78	0.79	0.79	0.79	0.79	0.79
	82	.67	.69	.71	.73	.76	.76	.77	.77	.78	.78	.79	.80	.80	.81	.82	.82	.83	.83	.84	.84	.84
	83	.72	.74	.76	.78	.81	.81	.81	.82	.82	.82	.83	.84	.85	.86	.87	.87	.87	.87	.87	.87	.88
	84	.77	.79	.81	.83	.86	.86	.86	.86	.87	.87	.88	.89	.90	.91	.92	.92	.93	.93	.93	.93	.94
	85	.83	.85	.87	.89	.91	.91	.92	.92	.93	.93	.94	.95	.96	.97	.98	.98	.98	.99	.99	.99	.99
	86	.88	.90	.92	.94	.96	.96	.97	.98	.98	.99	1.00	1.01	1.02	1.02	1.03	1.03	1.04	1.04	1.04	1.05	1.05
	87	.94	.96	.98	.99	1.01	1.02	1.03	1.03	1.04	1.05	1.05	1.06	1.07	1.07	1.08	1.08	1.09	1.10	1.10	1.11	1.11
	88	.99	1.01	1.03	1.04	1.06	1.07	1.08	1.09	1.10	1.11	1.11	1.12	1.12	1.13	1.13	1.14	1.15	1.15	1.16	1.17	1.17
	89	1.05	1.07	1.09	1.10	1.11	1.15	1.13	1.14	1.15	1.16	1.16	1.17	1.17	1.18	1.18	1.19	1.21	1.21	1.22	1.23	1.23
	90	1.11	1.13	1.15	1.16	1.17	1.18	1.19	1.20	1.21	1.22	1.22	1.23	1.23	1.24	1.24	1.25	1.27	1.28	1.29	1.30	1.30
	91	1.15	1.17	1.19	1.20	1.21	1.22	1.23	1.24	1.26	1.27	1.27	1.27	1.28	1.28	1.29	1.30	1.32	1.33	1.34	1.35	1.36
	92	1.20	1.22	1.24	1.25	1.26	1.27	1.28	1.30	1.31	1.32	1.32	1.33	1.33	1.34	1.34	1.35	1.37	1.38	1.39	1.40	1.41
	93	1.24	1.26	1.28	1.29	1.31	1.32	1.33	1.34	1.36	1.37	1.37	1.38	1.39	1.39	1.40	1.40	1.42	1.43	1.44	1.44	1.45
	94	1.29	1.31	1.33	1.35	1.36	1.37	1.39	1.40	1.41	1.42	1.42	1.43	1.44	1.44	1.45	1.45	1.47	1.47	1.48	1.49	1.50
	95	1.33	1.35	1.37	1.39	1.41	1.42	1.44	1.45	1.46	1.47	1.47	1.48	1.49	1.49	1.50	1.50	1.51	1.52	1.53	1.54	1.54
	96	1.38	1.40	1.42	1.44	1.46	1.47	1.49	1.50	1.51	1.52	1.52	1.53	1.54	1.54	1.55	1.55	1.56	1.57	1.57	1.58	1.58
	97	1.42	1.44	1.46	1.48	1.51	1.52	1.54	1.55	1.56	1.57	1.57	1.58	1.59	1.59	1.60	1.60	1.62	1.62	1.63	1.63	1.64
	98	1.47	1.49	1.51	1.54	1.56	1.57	1.58	1.59	1.60	1.62	1.63	1.63	1.64	1.64	1.65	1.65	1.66	1.67	1.67	1.68	1.68
	99	1.51	1.53	1.55	1.58	1.61	1.62	1.63	1.64	1.66	1.67	1.68	1.68	1.69	1.70	1.71	1.71	1.73	1.73	1.73	1.73	1.74
	100	1.55	1.57	1.59	1.62	1.66	1.67	1.68	1.69	1.72	1.72	1.73	1.73	1.74	1.76	1.77	1.77	1.78	1.79	1.79	1.79	1.80

N. B.—The correction for indications between 21° and 30° Balling and 50° and 70° F. temperature may be made by adding 0.005 to each correction given in the above table, column 21°, for each increase in degree Balling. For instance, 25° Balling 70° F., correction to be added to column 21° and 70°, 0.02+0.38=0.40.

TABLE II.—*Percentage of alcohol by weight and by volume.*

[Recalculated from Gilpin's, Drinkwater's, Squibb's, etc., determination, by EDGAR RICHARDS.]

Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.
1.00000	**0.00**	**0.00**	**0.99629**	**2.50**	**1.99**	**0.99281**	**5.00**	**4.00**	**0.98959**	**7.50**	**6.02**
0.99992	.05	.04	622	.55	2.03	274	.05	.04	953	.55	.06
984	.10	.08	615	.60	.07	268	.10	.08	947	.60	.10
976	.15	.12	607	.65	.11	261	.15	.12	940	.65	.14
968	.20	.16	600	.70	.15	255	.20	.16	934	.70	.18
961	.25	.20	593	.75	.19	248	.25	.20	928	.75	.22
953	.30	.24	586	.80	.23	241	.30	.24	922	.80	.26
945	.35	.28	579	.85	.27	235	.35	.28	916	.85	.30
937	.40	.32	571	.90	.31	228	.40	.32	909	.90	.34
930	.45	.36	564	.95	.35	222	.45	.36	903	.95	.38
.99923	**0.50**	**0.40**	**.99557**	**3.00**	**2.39**	**.99215**	**5.50**	**4.40**	**.98897**	**8.00**	**6.42**
915	.55	.44	550	.05	.43	208	.55	.44	891	.05	.46
907	.60	.48	543	.10	.47	202	.60	.48	885	.10	.50
900	.65	.52	536	.15	.51	195	.65	.52	879	.15	.54
892	.70	.56	529	.20	.55	189	.70	.56	873	.20	.58
884	.75	.60	522	.25	.59	182	.75	.60	867	.25	.62
877	.80	.64	515	.30	.64	175	.80	.64	861	.30	.67
869	.85	.67	508	.35	.68	169	.85	.68	855	.35	.71
861	.90	.71	501	.40	.72	162	.90	.72	849	.40	.75
854	.95	.75	494	.45	.76	156	.95	.76	843	.45	.79
.99849	**1.00**	**0.79**	**.99487**	**3.50**	**2.80**	**.99149**	**6.00**	**4.80**	**.98837**	**8.50**	**6.83**
842	.05	.83	480	.55	.84	143	.05	.84	831	.55	.87
834	.10	.87	473	.60	.88	136	.10	.88	825	.60	.91
827	.15	.91	466	.65	.92	130	.15	.92	819	.65	.95
819	.20	.95	459	.70	.96	123	.20	.96	813	.70	.99
812	.25	.99	452	.75	3.00	117	.25	5.00	807	.75	7.03
805	.30	1.03	445	.80	.04	111	.30	.05	801	.80	.07
797	.35	.07	438	.85	.08	104	.35	.09	795	.85	.11
790	.40	.11	431	.90	.12	098	.40	.13	789	.90	.15
782	.45	.15	424	.95	.16	091	.45	.17	783	.95	.19
.99775	**1.50**	**1.19**	**.99417**	**4.00**	**3.20**	**.99085**	**6.50**	**5.21**	**.98777**	**9.00**	**7.23**
768	.55	.23	410	.05	.24	079	.55	.25	771	.05	.27
760	.60	.27	403	.10	.28	072	.60	.29	765	.10	.31
753	.65	.31	397	.15	.32	066	.65	.33	759	.15	.35
745	.70	.35	390	.20	.36	059	.70	.37	754	.20	.39
738	.75	.39	383	.25	.40	053	.75	.41	748	.25	.43
731	.80	.43	376	.30	.44	047	.80	.45	742	.30	.48
723	.85	.47	369	.35	.48	040	.85	.49	736	.35	.52
716	.90	.51	363	.40	.52	034	.90	.53	730	.40	.56
708	.95	.55	356	.45	.56	027	.95	.57	724	.45	.60
.99701	**2.00**	**1.59**	**.99349**	**4.50**	**3.60**	**.99021**	**7.00**	**5.61**	**.98719**	**9.50**	**7.64**
694	.05	.63	342	.55	.64	015	.05	.65	713	.55	.68
687	.10	.67	335	.60	.68	009	.10	.69	707	.60	.72
679	.15	.71	329	.65	.72	002	.15	.73	701	.65	.76
672	.20	.75	322	.70	.76	.98996	.20	.77	695	.70	.80
665	.25	.79	315	.75	.80	990	.25	.81	689	.75	.84
658	.30	.83	308	.80	.84	984	.30	.86	683	.80	.88
651	.35	.87	301	.85	.88	978	.35	.90	678	.85	.92
643	.40	.91	295	.90	.92	971	.40	.94	672	.90	.96
636	.45	.95	288	.95	.96	965	.45	.98	666	.95	8.00

Table II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.
0.98660	**10.00**	**8.04**	**0.98381**	**12.50**	**10.08**	**0.98114**	**15.00**	**12.13**	**0.97859**	**17.50**	**14.19**
654	.05	.08	375	.55	.12	108	.05	.17	853	.55	.23
649	.10	.12	370	.60	.16	104	.10	.21	848	.60	.27
643	.15	.16	364	.65	.20	099	.15	.25	843	.65	.31
637	.20	.20	359	.70	.24	093	.20	.29	838	.70	.35
632	.25	.24	353	.75	.28	088	.25	.33	833	.75	.40
626	.30	.29	348	.80	.33	083	.30	.38	828	.80	.44
620	.35	.33	342	.85	.37	078	.35	.42	823	.85	.48
614	.40	.37	337	.90	.41	073	.40	.46	818	.90	.52
609	.45	.41	331	.95	.45	068	.45	.50	813	.95	.56
.98603	**10.50**	**8.45**	**.98326**	**13.00**	**10.49**	**.98063**	**15.50**	**12.54**	**.97808**	**18.00**	**14.60**
597	.55	.49	321	.05	.53	057	.55	.58	803	.05	.64
592	.60	.53	315	.10	.57	052	.60	.62	798	.10	.68
586	.65	.57	310	.15	.61	047	.65	.66	793	.15	.73
580	.70	.61	305	.20	.65	042	.70	.70	788	.20	.77
575	.75	.65	299	.25	.69	037	.75	.75	783	.25	.81
569	.80	.70	294	.30	.74	032	.80	.79	778	.30	.85
563	.85	.74	289	.35	.78	026	.85	.83	773	.35	.89
557	.90	.78	283	.40	.82	021	.90	.87	768	.40	.94
552	.95	.82	278	.45	.86	016	.95	.91	763	.45	.98
.98546	**11.00**	**8.86**	**.98273**	**13.50**	**10.90**	**.98011**	**16.00**	**12.95**	**.97758**	**18.50**	**15.02**
540	.05	.90	267	.55	.94	005	.05	.99	753	.55	.06
535	.10	.94	262	.60	.98	001	.10	13.03	748	.60	.10
529	.15	.98	256	.65	11.02	.97996	.15	.08	743	.65	.14
524	.20	9.02	251	.70	.06	991	.20	.12	738	.70	.18
518	.25	.07	246	.75	.11	986	.25	.16	733	.75	.22
513	.30	.11	240	.80	.15	980	.30	.20	728	.80	.27
507	.35	.15	235	.85	.19	975	.35	.24	723	.85	.31
502	.40	.19	230	.90	.23	970	.40	.29	718	.90	.38
496	.45	.23	224	.95	.27	965	.45	.33	713	.95	.39
.98491	**11.50**	**9.27**	**.98219**	**14.00**	**11.31**	**.97960**	**16.50**	**13.37**	**.97708**	**19.00**	**15.43**
485	.55	.31	214	.05	.35	955	.55	.41	703	.05	.47
479	.60	.35	209	.10	.39	950	.60	.45	698	.10	.51
474	.65	.39	203	.15	.43	945	.65	.49	693	.15	.55
468	.70	.43	198	.20	.47	940	.70	.53	688	.20	.59
463	.75	.47	193	.25	.52	935	.75	.57	683	.25	.63
457	.80	.51	188	.30	.56	929	.80	.62	678	.30	.68
452	.85	.55	182	.35	.60	924	.85	.66	673	.35	.72
446	.90	.59	177	.40	.64	919	.90	.70	668	.40	.76
441	.95	.63	172	.45	.68	914	.95	.74	663	.45	.80
.98435	**12.01**	**9.67**	**.98167**	**14.50**	**11.72**	**.97909**	**17.00**	**13.78**	**.97658**	**19.50**	**15.84**
430	.05	.71	161	.55	.76	904	.05	.82	653	.55	.88
424	.10	.75	156	.60	.80	899	.10	.86	648	.60	.93
419	.15	.79	151	.65	.84	894	.15	.90	643	.65	.97
413	.20	.83	146	.70	.88	889	.20	.94	638	.70	16.01
408	.25	.87	140	.75	.93	884	.25	.98	633	.75	.05
402	.30	.92	135	.80	.97	879	.30	14.03	628	.80	.09
397	.35	.96	130	.85	12.01	874	.35	.07	623	.85	.14
391	.40	10.00	125	.90	.05	869	.40	.11	618	.90	.18
386	.45	.04	119	.95	.09	864	.45	.15	613	.95	.22

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
0.97608	**20.00**	**16.26**	**0.97355**	**22.50**	**18.34**	**0.97097**	**25.00**	**20.43**	**0.96828**	**27.50**	**22.54**
603	.05	.30	350	.55	.38	092	.05	.47	822	.55	.58
598	.10	.34	345	.60	.42	086	.10	.51	816	.60	.62
593	.15	.38	340	.65	.47	081	.15	.56	811	.65	.67
588	.20	.42	335	.70	.51	076	.20	.60	805	.70	.71
583	.25	.46	330	.75	.55	071	.25	.64	800	.75	.75
578	.30	.51	324	.80	.59	065	.30	.68	794	.80	.79
573	.35	.58	319	.85	.63	060	.35	.72	789	.85	.83
568	.40	.59	314	.90	.68	055	.40	.77	783	.90	.88
563	.45	.63	309	.95	.72	049	.45	.81	778	.95	.92
.97558	**20.50**	**16.67**	**.97304**	**23.00**	**18.76**	**.97044**	**25.50**	**20.85**	**.96772**	**28.00**	**22.96**
552	.55	.71	299	.05	.80	039	.55	.89	766	.05	23.00
547	.60	.75	294	.10	.84	033	.60	.93	761	.10	.04
542	.65	.80	289	.15	.88	028	.65	.98	755	.15	.09
537	.70	.84	283	.20	.92	023	.70	21.02	749	.20	.13
532	.75	.88	278	.25	.96	018	.75	.06	744	.25	.17
527	.80	.92	273	.30	19.01	012	.80	.10	738	.30	.21
522	.85	.96	268	.35	.05	007	.85	.14	732	.35	.25
517	.90	17.01	263	.40	.09	001	.90	.19	726	.40	.30
512	.95	.05	258	.45	.13	.96996	.95	.23	721	.45	.34
.97507	**21.00**	**17.09**	**.97253**	**23.50**	**19.17**	**.96991**	**26.00**	**21.27**	**.96715**	**28.50**	**23.38**
502	.05	.13	247	.55	.21	986	.05	.31	709	.55	.42
497	.10	.17	242	.60	.25	980	.10	.35	704	.60	.47
492	.15	.22	237	.65	.30	975	.15	.40	698	.65	.51
487	.20	.26	232	.70	.34	969	.20	.44	692	.70	.55
482	.25	.30	227	.75	.38	964	.25	.48	687	.75	.60
477	.30	.34	222	.80	.42	959	.30	.52	681	.80	.64
472	.35	.38	216	.85	.46	953	.35	.56	675	.85	.68
467	.40	.43	211	.90	.51	949	.40	.61	669	.90	.72
462	.45	.47	206	.95	.55	942	.45	.65	664	.95	.77
.97457	**21.50**	**17.51**	**.97201**	**24.00**	**19.59**	**.96937**	**26.50**	**21.69**	**.96658**	**29.00**	**23.81**
451	.55	.55	196	.05	.63	932	.55	.73	652	.05	.85
446	.60	.59	191	.10	.67	926	.60	.77	646	.10	.89
441	.65	.63	185	.15	.72	921	.65	.82	640	.15	.94
436	.70	.67	180	.20	.76	915	.70	.86	635	.20	.98
431	.75	.71	175	.25	.80	910	.75	.90	629	.25	24.02
426	.80	.76	170	.30	.84	905	.80	.94	623	.30	.06
421	.85	.80	165	.35	.88	899	.85	.98	617	.35	.10
416	.90	.84	159	.40	.93	894	.90	22.03	611	.40	.15
411	.95	.88	154	.45	.97	888	.95	.07	605	.45	.19
.97406	**22.00**	**17.92**	**.97149**	**24.50**	**20.01**	**.96883**	**27.00**	**22.11**	**.96600**	**29.50**	**24.23**
401	.05	.96	144	.55	.05	877	.05	.15	594	.55	.27
396	.10	18.00	139	.60	.09	872	.10	.20	587	.60	.32
391	.15	.05	133	.65	.14	866	.15	.24	582	.65	.36
386	.20	.09	128	.70	.18	861	.20	.28	576	.70	.40
381	.25	.13	123	.75	.22	855	.25	.33	570	.75	.45
375	.30	.17	118	.80	.26	850	.30	.37	564	.80	.49
370	.35	.21	113	.85	.30	844	.35	.41	559	.85	.53
365	.40	.26	107	.90	.35	839	.40	.45	553	.90	.57
360	.45	.30	102	.95	.39	833	.45	.50	547	.95	.62

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
0.96541	**30.00**	**24.66**	**0.96235**	**32.50**	**26.80**	**0.95910**	**35.00**	**28.96**	**0.95560**	**37.50**	**31.14**
535	.05	.70	229	.55	.84	903	.05	29.00	552	.55	.18
529	.10	.74	222	.60	.89	896	.10	.05	545	.60	.23
523	.15	.79	216	.65	.93	889	.15	.09	538	.65	.27
517	.20	.83	210	.70	.97	883	.20	.13	531	.70	.32
511	.25	.87	204	.75	27.02	876	.25	.18	523	.75	.36
505	.30	.91	197	.80	.06	869	.30	.22	516	.80	.40
499	.35	.95	191	.85	.10	862	.35	.26	509	.85	.45
493	.40	25.00	185	.90	.14	855	.40	.30	502	.90	.49
487	.45	.04	178	.95	.19	848	.45	.35	494	.95	.54
.96181	**30.50**	**25.08**	**.96172**	**33.00**	**27.23**	**.95842**	**35.50**	**29.39**	**.95487**	**38.00**	**31.58**
475	.55	.12	166	.05	.27	835	.55	.43	480	.05	.63
469	.60	.17	159	.10	.32	828	.60	.48	472	.10	.67
463	.65	.21	153	.15	.36	821	.65	.52	465	.15	.72
457	.70	.25	146	.20	.40	814	.70	.57	457	.20	.76
451	.75	.30	140	.25	.45	807	.75	.61	450	.25	.81
445	.80	.34	133	.30	.49	800	.80	.65	442	.30	.85
439	.85	.38	127	.35	.53	794	.85	.70	435	.35	.90
433	.90	.42	120	.40	.57	787	.90	.74	427	.40	.94
427	.95	.47	114	.45	.62	780	.95	.79	420	.45	.99
.96421	**31.00**	**25.51**	**.96108**	**33.50**	**27.66**	**.95773**	**36.00**	**29.83**	**.95413**	**8.50**	**32.03**
415	.05	.55	101	.55	.70	766	.05	.87	405	.55	.07
409	.10	.60	095	.60	.75	759	.10	.92	398	.60	.12
403	.15	.64	088	.65	.79	752	.15	.96	390	.65	.16
396	.20	.68	082	.70	.83	745	.20	30.00	383	.70	.20
390	.25	.73	075	.75	.88	738	.25	.05	375	.75	.25
384	.30	.77	069	.80	.92	731	.30	.09	368	.80	.29
378	.35	.81	062	.85	.96	724	.35	.13	360	.85	.33
372	.40	.85	056	.90	28.00	717	.40	.17	353	.90	.37
366	.45	.90	049	.95	.05	710	.45	.22	345	.95	.42
.96360	**31.50**	**25.94**	**.96043**	**34.00**	**28.09**	**.95703**	**36.50**	**30.26**	**.95338**	**39.00**	**32.46**
353	.55	.98	036	.05	.13	695	.55	.30	330	.05	.50
347	.60	26.03	030	.10	.18	688	.60	.35	323	.10	.55
341	.65	.07	023	.15	.22	681	.65	.39	315	.15	.59
335	.70	.11	016	.20	.26	674	.70	.44	307	.20	.64
329	.75	.16	010	.25	.31	667	.75	.48	300	.25	.68
323	.80	.20	003	.30	.35	660	.80	.52	292	.30	.72
316	.85	.24	.95996	.35	.39	653	.85	.57	284	.35	.77
310	.90	.28	990	.40	.43	646	.90	.61	277	.40	.81
304	.95	.33	983	.45	.48	639	.95	.66	269	.45	.86
.96298	**32.00**	**26.37**	**.95977**	**34.50**	**28.52**	**.95632**	**37.00**	**30.70**	**.95262**	**39.50**	**32.90**
292	.05	.41	970	.55	.56	625	.05	.74	254	.55	.95
285	.10	.46	963	.60	.61	618	.10	.79	246	.60	.99
279	.15	.50	957	.65	.65	610	.15	.83	239	.65	33.04
273	.20	.54	950	.70	.70	603	.20	.88	231	.70	.08
267	.25	.59	943	.75	.74	596	.25	.92	223	.75	.13
260	.30	.63	937	.80	.78	589	.30	.96	216	.80	.17
254	.35	.67	930	.85	.83	581	.35	31.01	208	.85	.22
248	.40	.71	923	.90	.87	574	.40	.05	200	.90	.27
241	.45	.76	917	.95	.92	567	.45	.10	193	.95	.31

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60^\circ}{60^\circ}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight
0.95185	**40.00**	**33.35**	**0.94786**	**42.50**	**35.58**	**0.94364**	**45.00**	**37.84**	**0.93916**	**47.50**	**40.13**
177	.05	.39	778	.55	.63	355	.05	.89	906	.55	.18
169	.10	.44	770	.60	.67	346	.10	.93	898	.60	.22
161	.15	.48	761	.65	.72	338	.15	.98	888	.65	.27
154	.20	.53	753	.70	.76	329	.20	38.02	879	.70	.32
146	.25	.57	745	.75	.81	320	.25	.07	870	.75	.37
138	.30	.61	737	.80	.85	311	.30	.12	861	.80	.41
130	.35	.66	729	.85	.90	302	.35	.16	852	.85	.46
122	.40	.70	720	.90	.94	294	.40	.21	842	.90	.51
114	.45	.75	712	.95	.99	285	.45	.25	833	.95	.55
.95107	**40.50**	**33.79**	**.94704**	**43.00**	**36.03**	**.94276**	**45.50**	**38.30**	**.93824**	**48.00**	**40.60**
099	.55	.84	696	.05	.08	267	.55	.35	815	.05	.65
091	.60	.88	687	.10	.12	258	.60	.39	805	.10	.69
083	.65	.93	679	.15	.17	250	.65	.44	796	.15	.74
075	.70	.97	670	.20	.21	241	.70	.48	786	.20	.78
067	.75	34.02	662	.25	.23	232	.75	.53	777	.25	.83
059	.80	.06	654	.30	.30	223	.80	.57	768	.30	.88
052	.85	.11	645	.35	.35	214	.85	.62	758	.35	.92
044	.90	.15	637	.40	.39	206	.90	.66	749	.40	.97
036	.95	.20	628	.45	.44	197	.95	.71	739	.45	41.01
.95028	**41.00**	**34.24**	**.94620**	**43.50**	**36.48**	**.94188**	**46.00**	**38.75**	**.93730**	**48.50**	**41.06**
020	.05	.28	612	.55	.53	179	.05	.80	721	.55	.11
012	.10	.33	603	.60	.57	170	.10	.84	711	.60	.15
004	.15	.37	595	.65	.62	161	.15	.89	702	.65	.20
.94996	.20	.42	586	.70	.66	152	.20	.93	692	.70	.24
988	.25	.46	578	.75	.71	143	.25	.98	683	.75	.29
980	.30	.50	570	.80	.75	134	.30	39.03	679	.80	.34
972	.35	.55	561	.85	.80	125	.35	.07	664	.85	.38
964	.40	.59	553	.90	.84	116	.40	.12	655	.90	.43
956	.45	.64	544	.95	.89	107	.45	.16	645	.95	.47
.94948	**41.50**	**34.68**	**.94536**	**44.00**	**36.93**	**.94098**	**46.50**	**39.21**	**.93636**	**49.00**	**41.52**
940	.55	.73	527	.05	.98	089	.55	.26	626	.05	.57
932	.60	.77	519	.10	37.02	080	.60	.30	617	.10	.61
924	.65	.82	510	.15	.07	071	.65	.35	607	.15	.66
916	.70	.86	502	.20	.11	062	.70	.39	598	.20	.71
908	.75	.91	493	.25	.16	053	.75	.44	588	.25	.76
900	.80	.95	484	.30	.21	044	.80	.49	578	.30	.80
892	.85	35.00	476	.35	.25	035	.85	.53	569	.35	.85
884	.90	.04	467	.40	.30	026	.90	.58	559	.40	.90
876	.95	.09	459	.45	.34	017	95	.62	550	.45	.94
.94868	**42.00**	**35.13**	**.94450**	**44.50**	**37.39**	**.94008**	**47.00**	**39.67**	**.93540**	**49.50**	**41.99**
860	.05	.18	441	.55	.44	.93999	.05	.72	530	.55	42.04
852	.10	.22	433	.60	.48	990	.10	.76	521	.60	.08
843	.15	.27	424	.65	.53	980	.15	.81	511	.65	.13
835	.20	.31	416	.70	.57	971	.20	.85	502	.70	.18
827	.25	.36	407	.75	.62	962	.25	.90	492	.75	.23
810	.30	.40	398	.80	.66	953	.30	.95	482	.80	.27
811	.35	.45	390	.85	.71	944	.35	.99	473	.85	.32
802	.40	.49	381	.90	.76	934	.40	40.04	463	.90	.37
794	.45	.54	373	.95	.80	925	.45	.08	454	.95	.41

METHODS FOR SOIL AND ASH ANALYSIS.

SOIL.

PREPARATION OF THE SAMPLE.

Five hundred grams or more of the air-dried soil, which may be either the original soil or that which has been passed through a sieve of coarser mesh, are sifted upon a sieve with circular openings one-half millimeter in diameter, rubbing, if necessary, with a rubber pestle in a mortar until the fine earth has been separated as completely as possible from the particles that are too coarse to pass the sieve. The fine earth is thoroughly mixed and preserved in a tightly stoppered bottle from which the portions for analysis are weighed out.

The coarse part is weighed and may be subjected to further examination. (See Bulletin 38, Div. Chem., pp. 65, 75, 200.)

It may sometimes be necessary to wash the soil through the one-half millimeter sieve with water, in which case proceed as directed on pages 65 and 75 of the above bulletin. The use of water is to be avoided whenever possible.

MOISTURE.

Heat 2 to 5 grams of the air-dried soil in a flat-bottom, tared platinum dish for five hours in a water oven kept briskly boiling; cover the dish, cool in a desiccator, and weigh. Repeat the heating, cooling, and weighing at intervals of two hours till constant weight is found and estimate the moisture by the loss of weight. Weigh rapidly to avoid absorption of moisture from the air. An air bath must not be used in this determination.

VOLATILE MATTER.

The platinum dish and soil used to determine moisture is used also to determine volatile matter. Heat the dish and dry soil to full redness until all organic matter is burned away. If the soil contained appreciable quantities of carbonates, the contents of the dish, after cooling, are to be moistened with a few drops of a saturated solution of ammonium carbonate, dried and heated to dull redness to expel salts of ammonia, cooled in the desiccator, and weighed. The loss in weight represents the organic matter, water of combination, salts of ammonia, etc.

ACID SOLUBLE MATERIALS.

In the following scheme for soil analysis it is intended to use the air-dried soil from the sample bottle for each separate investigation. The determination of moisture, made once for all on a separate portion of air-dried soil, will afford the datum for calculating the results of analysis upon the soil dried at the temperature of boiling water. It is not desirable to ignite the soil before analysis, or to heat it so as to change its chemical properties.

The acid digestion is to be performed in a flask so arranged that the evaporation of acid shall be reduced to a minimum, but under atmospheric pressure and at the temperature of boiling water. The digestion is easily accomplished in a flat-bottom conical flask of hard glass, carrying a stopper and hard-glass condensing tube at least 18 inches long. Where SO_3 is to be determined, a rubber stopper can not be used. A flask with ground-glass stopper, carrying a condensing tube, is useful in such cases.

The flask must be immersed in the water bath up to the neck, or at least to the level of the acid, and the water must be kept boiling continuously during the digestion.

In the following scheme 10 grams of soil are taken, this being a convenient quantity in most soils, in which the insoluble matter is about 80 per cent. If desired, a larger quantity of such soil may be taken, using a proportionately larger quantity of acid and making up the soil solution to a proportionately larger volume. In very sandy soils, where the proportion of insoluble matter is 90 per cent or more, 20 grams of soil are to be digested with 100 cc. of acid and the solution made up to 500 cc.; or a larger quantity may be used, preserving the same proportions. It is very important that the analyst assure himself of the purity of all the reagents to be used in the analysis of soils before beginning the work.

(1) *Acid digestion of the soil.*—Place 10 grams of the air-dried soil in a 150 to 200 cc. Bohemian glass flask, add 100 cc. of pure HCl of sp. gr. 1.115, insert the stopper with condensing tube, place in a water or steam bath and digest for ten hours continuously at the temperature of boiling water, shaking once each hour. Pour the clear liquid from the flask into a small beaker, wash the residue out of the flask with distilled water on a filter, adding the washings to the contents of the beaker. The residue, after washing until free of acid, is to be dried and ignited, as directed below. Add 1 or 2 cc. of nitric acid to the filtrate and evaporate to dryness on the water bath, finishing on a sand or air bath to complete dryness; take up with hot water and a few cubic centimeters of HCl and again evaporate to complete dryness. Take up as before, filter and wash thoroughly with cold water or with hot water slightly acidified at first with HCl. Cool and make up to 500 cc. This is solution A. The residue is to be added to the main residue and the whole ignited and weighed, giving the "insoluble matter."

(2) *Ferric oxid, alumina, and phosphoric acid.*—To 100 or 200 cc. of solution A, according to the probable amount of iron and alumina present, add ammonium hydroxid to slightly alkaline reaction to precipitate ferric and aluminic hydrates and phosphates. Expel the excess of ammonia by boiling, allow to settle, decant the clear solution through a filter; add to the flask 50 cc. of hot distilled water, boil, settle, and decant as before. After pouring off all the clear solution possible, dissolve the residue with a few drops of HCl and precipitate again with ammonium hydroxid exactly as before; transfer all the precipitate to the filter and wash with hot distilled water till the washings become free from chlorids. Save the filtrates and solutions which form solution B. Dry the filter and precipitate, transfer the precipitate to a tared platinum crucible, burn the filter and add the ash to the precipitate; ignite to bright redness, cool in a desiccator, and weigh. The increase of weight, minus the ash of filter and the phosphoric acid (found in a separate process), represents the weight of the ferric and aluminic oxids.

(3) *Manganese.*—Concentrate the filtrate and washings (solution B) to 100 cc. or less; add ammonium hydroxid to alkalinity; add bromin water and heat to boiling, keeping beaker covered with a watch crystal; as the bromin escapes the beaker is allowed to cool somewhat, more ammonia and bromin water being added and heated as before. This process is continued until the manganese is completely precipitated, which requires from fifteen to thirty minutes. The solution is then to be slightly acidified with a few drops of acetic acid and filtered while still boiling hot, the precipitate washed with hot water, dried, ignited, and weighed as Mn_3O_4.

(4) *Lime.*—If no manganese is precipitated, evaporate solution B or the filtrates and washings from (3) to about 50 cc., make slightly alkaline with ammonia and add, while still hot, $(NH_4)_2C_2O_4$ solution as long as any precipitation is produced, adding a few cubic centimeters in excess to convert the magnesia also into oxalate. Heat to boiling and let stand for a few minutes till the calcium oxalate settles clear, decant the clear solution on a filter, pour 15 or 20 cc. of hot distilled water on the precipitate and again decant the clear solution on the filter. Dissolve the precipitate in the beaker with a few drops of HCl, add a little water and reprecipitate, boiling hot, by adding ammonium hydroxid to slight alkalinity and a little $(NH_4)_2C_2O_4$ solution; filter through the same filter, transfer the precipitate to the filter, and wash it free from chlorids. Dry, ignite the precipitate over the blast lamp until it ceases to lose weight, weigh, and estimate as CaO.

(5) *Magnesia.*—Acidify the filtrate and washings from (4) slightly with HCl and concentrate to about 50 cc., place in a small Erlenmeyer flask or beaker, make

slightly alkaline with ammonium hydroxid and add a few cubic centimeters of Na_2HPO_4 solution sufficient to precipitate the magnesia; then add gradually 10 cc. strong ammonium hydroxid, cover closely to prevent escape of ammonia, and let stand in the cold. Filter after twelve hours, wash the precipitate free of chlorids, dry, burn at first at a moderate heat; finally igniting intensely and weigh $Mg_2P_2O_7$.

(6) *Ferric oxid.*—Evaporate 100 cc. of solution A with the addition of about 100 cc. of H_2SO_4 until all HCl is expelled; dilute with water, reduce with zinc and estimate ferric oxid by a standard solution of potassium permanganate. To prepare potassium permanganate solution, dissolve 3.156 grams of pure crystallized potassium permanganate in 2,000 cc. of distilled water, and preserve in a ground-glass-stoppered bottle, shielded from the light. Standardize this solution after it has stood twenty-four hours with pure ammonio-ferrous sulphate or oxalic acid or with metallic iron.

Instead of taking another portion of the solution, the weighed precipitate from (2) may be dissolved by digestion on the water bath in a covered beaker or flask with 10 to 20 cc. of a mixture of 1 part H_2SO_4 with 4 parts of water.

Deduct the per cent of ferric oxid obtained from the per cent of ferric oxid and alumina (2), with corrections for filter ash and phosphoric acid, to obtain the per cent of alumina.

(7) *Phosphoric acid.*—100 to 200 cc. of solution A are evaporated to about 25 or 30 cc. on the water bath; nearly neutralize with ammonium hydroxid, add about 10 grams pure crystallized ammonium nitrate, and then add gradually about 20 cc. molybdic solution and set in water bath at a temperature of 40° C. When the precipitate has settled sufficiently, draw out with a pipette about 5 cc. of the clear liquid and test it by allowing it to run into 5 cc. of warm molybdic solution. If any precipitate is produced, the test liquid is returned to the main portion and more molybdic solution is added and the operation repeated until all the P_2O_5 is precipitated. After standing eight to twelve hours at a temperature not above 40°, the ammonium phosphomolybdate is filtered off and the P_2O_5 determined as $Mg_2P_2O_7$ as usual. (See total phosphoric acid in fertilizers.) It is recommended to redissolve the magnesium ammonium phosphate precipitate in acetic acid, after it has been washed once or twice, and reprecipitate with ammonia and a fresh quantity of magnesia solution, of course giving the usual time for the separation of the precipitate. If there is any residue of phosphates remaining on dissolving the phosphomolybdate in ammonia, or the magnesium ammonium phosphate in acetic acid, this residue is to be dissolved in a little HCl, neutralized with ammonium hydroxid and precipitated with molybdic solution and the phosphomolybdate obtained added to the main quantity.

(8) *Sulphuric acid.*—Evaporate 100 to 200 cc. of solution A nearly to dryness on a water bath to expel the excess of acid; then add 50 cc. of distilled water; heat to boiling and add 2 or 3 cc. of a solution of barium chlorid, and continue the boiling for five minutes. When the precipitate is settled pour the liquid on a tared Gooch filter, treat the precipitate with 15 or 20 cc. of boiling water, and transfer the precipitate to the filter and wash with boiling water at first slightly acidified with a few drops of HCl, finally with pure water till the filtrate is free from chlorids. Dry the filter and ignite strongly. The increase in weight is barium sulphate, which multiplied by 0.34331 equals SO_3.

(9) *Potash and soda.*—Treat the filtrate from (8) with ammonium hydroxid exactly as in (2). Evaporate the filtrate and washings to dryness, heat below redness, until ammonia salts are expelled, dissolve in about 25 cc. of hot water, add 5 cc. of baryta water and heat to boiling; let settle a few minutes and test a little of the clear liquid with more baryta water to be sure that enough has been added. When no farther precipitate is produced, filter and wash thoroughly with hot water. Add ammonia and ammonium carbonate to complete the precipitation of the baryta, let stand a short time on the water bath, filter and wash the precipitate thoroughly with hot water, evaporate filtrate and washings to dryness in a porcelain dish, expel

ammonia salts again by heat below redness, take up with a little hot water, add a few drops of ammonium hydroxid and a drop or two of ammonium carbonate, let stand a few minutes on the water bath, filter into a tared platinum dish, evaporate to dryness on the water bath and heat to dull redness, until all ammonia salts are expelled and the residue is nearly or quite white. The heat must not be sufficient to fuse the residue. The weight of the residue represents potassium and sodium chlorids. Determine the potassium present with platinum chlorid in the usual manner. The sodium chlorid is obtained by subtracting potassium chlorid thus found from the total weight obtained above.

Instead of this, a fresh aliquot of solution A may be evaporated to dryness, redissolved in water and treated directly with milk of lime as in ash analysis, but without previous addition of $BaCl_2$.

(10) *Total alkalies.*—Determine in a separate portion of the soil by J. Lawrence Smith's method given in Crooke's Select Methods, second edition, pages 28–40. Or, preferably, determine by this method the alkalies in the insoluble residue from (1) and add the amount obtained from the HCl solution.

(11) *Other alkali metals.*—The salts of lithium, cesium, and rubidium, are occasionally found in very small amounts in soils. The agricultural uses of these salts are still in question, and their amount is too small to admit of quantitative estimation. A qualitative examination may be made by the spectroscope with the water soluble materials evaporated to dryness and dissolved with two or three drops of HCl or with the alkaline chlorids separated as in (9) or (10). Test by the spectroscope with a platinum wire in a Bunsen flame.

(12) *Siliceous residue.*—The siliceous residue from (1) may be analyzed by the usual methods for silicates. If it is desired to determine the SiO_2 soluble in alkalies, the residue must be dried at 100° and an aliquot taken before ignition for treatment with Na_2CO_3 solution as described under ash analysis. Another aliquot or the rest of the residue, will of course be ignited and weighed.

(13) *Total nitrogen.*—Is determined by the "Kjeldahl method modified to include nitrates" as described in the methods for determining nitrogen, using 14 grams of soil, 0.5 gram salicylic acid in 20 cc. H_2SO_4, and 2.5 grams sodium thiosulphate. When the digestion is completed, water is added and the solution filtered or decanted from the insoluble residue and the residue washed with more water. The filtrate and washings are treated in the usual manner for determination of the ammonia. Unless the amount of nitrates present is considerable it is not necessary to use the modified method. The amount of nitrate present in ordinary soils is so small that the simple Kjeldahl method, carried out as just described, omitting the salicylic acid and thiosulphate, is perfectly satisfactory.

(14) *Carbon dioxid.*—Determine as in ash analysis, using 5 to 50 grams of soil, according to the amount present.

STATEMENT OF RESULTS.

All results of soil analyses are to be calculated as per cent of the soil dried to constant weight in the water oven (*see* "Moisture") and are to be stated in the order given on page 82, of Bulletin 38, Division of Chemistry.

[In the absence of formal action by the Association upon certain points in soil analysis, the following suggestions are made by the Reporter.]

Sampling.—The soil sample to be taken as described in Bulletin 38, Division of Chemistry, page 200, having reference also to the fuller instructions given by Dr. Hilgard on page 61 of the same bulletin.

Mechanical analysis.—Use Hilgard's method as described in Bulletin 38, Division of Chemistry, page 62 *et. seq.*, as the standard method, or the beaker method of Osborne (Conn. State Sta., Ann. Report, 1886, p. 144) as an alternate method.

Water capacity.—Use the method described by Dr. Hilgard in Bulletin 38, Division of Chemistry, page 73.

Humus.—Use the Grandeau method as modified by Huston and McBryde (Bull. 38, Div. Chem., p. 84), or by Snyder (Jour. Am. Chem. Soc., XVI, 3).

Capillary power and water soluble materials.—Use the methods described in Bulletin 38, Division of Chemistry, page 201.

Hygroscopic coefficient.—Expose 5 to 10 grams of the fine earth, in a thin layer, for twenty-four hours, to an atmosphere saturated artificially with moisture and at a temperature of 20° C.; weigh covered and calculate the increase of weight, including the moisture already present as per cent of the soil dried in the water oven. (*See* "Moisture.")

ANALYSIS OF THE ASHES OF PLANTS.

PREPARATION OF THE ASH.

Before combustion the material must be thoroughly cleaned from all foreign matter, especially from adhering soil. The combustion should be carried on at a comparatively low temperature, never reaching a full red heat, because of the danger of volatilizing alkaline chlorids, etc., and of fusing the ash; nor in a strong draft of air, lest the lighter part of the ash be carried away. Combustion is best carried on in a flat platinum dish in a muffle. With substances rich in silica and alkalies it is better to first char the substance, wash with the distilled water to remove soluble salts, then dry and incinerate the residue. Evaporate the watery extract and add this to the rest of the ash. With substances rich in phosphates, e. g., seeds and animal substances, char the material and remove salts by acetic acid, decant the acetic solution, wash with distilled water, and then complete the combustion. Add the acetic solution and washings to the final ash, evaporate to dryness, and gently ignite the whole to decompose the acetates. In whatever way obtained the whole of the ash should be pulverized and intimately mixed while still warm, and preserved in a tight dry bottle for analysis. If necessary, dry thoroughly at low redness before bottling. It is intended that the preliminary preparation of the ash shall bring it to a perfectly dry condition, rendering a moisture determination unnecessary, and that the portions for analysis shall be weighed out from the prepared sample in this condition. As it is sometimes difficult or impossible to prepare an ash for analysis in large quantity, the following method has been condensed as much as possible so that a small amount of ash may be used. Where the ash is to be had in abundance, larger quantities and more portions may be used, but the proportion of acid to substance taken is to be preserved. Much latitude must be left to individual judgment in adapting the method to particular cases.

ANALYSIS OF THE ASH.

Five grams of the ash are treated in a beaker, covered with a watch glass, with 50 cc. HCl, sp. gr. 1.115 and digested on the water bath until all effervescence has ceased. The cover is then removed and the liquid evaporated to complete dryness to render silica insoluble. The residue is moistened with 2 or 3 cc. of HCl and taken up with about 50 cc. water, allowed to stand on the water bath a few minutes and filtered through a C. S. & S. "hardened" filter, and thoroughly washed. The filtrate and washings are made up to 250 cc. for analysis. This is Solution A.

(1) *Carbon, sand, and silica.*—The residue is washed from the filter (which may be used again) as completely as possible into a platinum dish and boiled about five minutes with about 20 cc. of a saturated solution of pure sodium carbonate, a few drops of pure NaOH solution are added and the liquid allowed to settle, and decanted through a tared Gooch filter. The residue is boiled with sodium carbonate solution and decanted as before a second and a third time, and finally brought upon the filter and thoroughly washed, first with hot water, then with a little dilute HCl, and finally with hot water until free of chlorids. The residue in the Gooch filter is dried at 110° to constant weight, giving the carbon and sand. It

is then incinerated and the weight of the sand determined, the difference giving the carbon. It is advisable to examine the sand with a microscope to determine if it is really sand. The alkaline filtrate and washings from the carbon and sand are acidified with HCl, evaporated to dryness, and the silica separated and determined in the usual way.

Instead of determining soluble SiO_2 directly from the Na_2CO_3 solution, as above, another portion of the ash may be treated with HCl and evaporated to dryness as before described, filtered on an ordinary filter, washed, burned, and weighed, giving the weight of silica plus sand, from which the weight of sand is deducted to obtain soluble SiO_2. It is inadmissible to separate the soluble SiO_2 from the residue after it has been ignited.

(2) *Manganese, lime, and magnesia.*—50 to 100 cc. of solution A are neutralized with $(NH_4)HO$ and a gram or two of sodium acetate and a few drops of acetic acid are added; boil for one or two minutes, filter and wash the precipitate thoroughly with boiling water. Evaporate the filtrate and washings to about 50 cc., filter out any slight additional precipitate that may form, and determine Mn, Ca, and Mg exactly as described in the analysis of soils. If difficulty is found in filtering the iron and alumina phosphates above, some Fe_2Cl_6 solution may be added before precipitating with sodium acetate.

(3) *Phosphoric acid.*—25 to 50 cc. of solution A are neutralized with $(NH_4)HO$, nitrate of ammonia added and precipitated with molybdic solution exactly as described in the methods for total P_2O_5 in fertilizers.

(4) *Sulphuric acid and alkalis.*—25 to 50 cc. of solution A are heated to boiling and $BaCl_2$ solution added in small quantity until no further precipitate is produced. Let stand on the water bath until clear and filter the $BaSO_4$ on a tared Gooch filter, wash thoroughly with hot water, at first slightly acidified with HCl, finally with pure water until free of chlorids, dry, and ignite the $BaSO_4$ and weigh.

Evaporate the filtrate and washings in a porcelain dish to dryness, redissolve in about 50 cc. water and add milk of lime, or $Ba(OH)_2$ solution, which must be perfectly free from alkalis, until no further precipitation is produced and it is evident there is an excess of $Ca(OH)_2$ or $Ba(OH)_2$ present, boil two or three minutes, filter hot, and wash thoroughly with boiling water, precipitate the lime and baryta from the filtrate with $(NH)_4HO$ and $(NH_4)_2CO_3$, filter, evaporate the filtrate to dryness in a porcelain dish, and drive off the ammonia salts by heat below redness. When cold, redissolve in 15 or 20 cc. water, precipitate again with a few drops of $(NH_4)HO$ and $(NH_4)_2CO_3$ solution, let stand a few minutes on the water bath and filter into a tared platinum dish and evaporate to dryness, expel the ammonia salts by heating to just perceptible dull redness, weigh the KCl and NaCl obtained and determine the KCl with platinum chlorid as usual.

Instead of the above method, the filtrate from the $BaSO_4$ may be treated exactly as described for soil analysis.

(5) *Carbonic acid.*—Use 1 to 5 grams of ash in any of the usual forms of apparatus, determining the CO_2 evolved either by increase of weight of potash bulbs or loss of weight of the apparatus. The former is preferable.

(6) *Chlorin.*—Determine as AgCl, either gravimetrically or by one of the standard volumetric processes, in a nitric acid or water solution of the ash. Nitric acid may be used in (5) and the solution taken for this purpose.

METHOD FOR ANALYSIS OF TANNING MATERIALS.

Testing the hide powder.—Shake 10 grams of the hide powder three times for five minutes with water, using 200 cc. each time. Evaporate 100 cc. of the final filtrate and if the residue amounts to more than 0.01 gram the hide powder should be rejected.

Moisture in the sample.—Dry 2 grams in a small dish at 100° C. until the weight becomes constant.

PROVISIONAL METHOD.

(1) Use such an amount of extract that 100 cc. of the solution shall contain about 1 gram of dry solids.

(2) Add to the extract taken about 200 cc. of water at 80° C., stir until dissolved, transfer to a graduated flask, washing out the beaker with warm water, cool, and make up to the mark. Shake thoroughly, immediately take 100 cc., evaporate, dry to constant weight at 100° C., and weigh.

Result is total solids.

(3) Now filter the remainder of the solution through thick filter paper, 9 cm. in diameter (S. and S. sirup-filter paper is recommended), using a teaspoonful of kaolin if necessary. Take 100 cc. of the filtrate, evaporate, and dry as before.

Result is soluble solids.

(4) Take 10 grams of hide powder, shake three times for five minutes with water, using 200 cc. each time. Strain through well-washed muslin or linen and squeeze out in a copying or other suitable press. Determine the moisture in about 3 grams of the wet hide powder by drying at about 60° C. to constant weight. Weigh the remaining powder and divide into three equal parts.

(5) Take 150 cc. of the filtered tannin liquor and shake up with the first portion for about five minutes. Strain through muslin or linen. To the strained liquid add the second portion of hide powder and shake again for about five minutes. Strain as before and shake up with the last portion for about eight minutes. Now filter through sirup-filter paper, pouring the liquor back until a clear filtrate is obtained. Take 100 cc., evaporate, and dry as before. The difference between the soluble solids and the final residue is the amount taken up by the hide powder. The result must be corrected for the water retained by the hide powder.

It is absolutely necessary to use some form of mechanical shaker, as shaking by hand is not intense enough to remove all the tannin. The machine known as the "milkshake" is recommended.

The hide powder must be kept in a dry place and tested once a month.

The above method is applicable to barks, extracts, and sweet liquors.

The dishes in which the drying is conducted should be covered while they are in the oven, so as to prevent loss by popping.

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CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects shall be (1) to secure uniformity and accuracy of the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership, and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members, and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also officiate as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a reporter and associate reporter for each of the subjects to be considered by the association.

It shall be the duty of these reporters to prepare and distribute samples and standard reagents to members of the association and others desiring the same; to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by a vote of two-thirds of the members present entitled to vote.

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